

Performance Evaluation of Full-Scale
Hazardous Waste Incinerators. Volume 4
Appendices C through J

Midwest Research Inst., Kansas City, MO

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PERFORMANCE EVALUATION OF FULL-SCALE
HAZARDOUS WASTE INCINERATORS

VOLUME IV

APPENDICES C THROUGH J

by

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16. ABSTRACT This report is Volume IV, Appendices C through J, of a series of extensive performance data EPA obtained through eight (8) field tests of actual industrial/commercial incinerators, was prepared in response to the Agency's need to conduct a regulatory impact analysis (RIA) for hazardous waste incinerators to determine the need, adequacy, and economic feasibility of current RCRA incinerator regulations. Tests involved trial burn-type testing at each plant during typical albeit closely monitored daily operations on wastes typical of their routine disposal function. Most incinerators were found to be closely approaching or performing within RCRA requirements for destruction of compounds, although some facilities showed substandard results in other areas.					
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FOREWORD

When energy and material resources are extracted, processed, converted, and used, the related pollutional impacts on our environment and even on our health often require that new and increasingly more efficient pollution control methods be used. The Industrial Environmental Research Laboratory-Cincinnati (IERL-Ci) assists in developing and demonstrating new and improved methodologies that will meet these needs both efficiently and economically.

This report is one of a five-volume series of reports presenting the recent testing and evaluation results of the performance of eight (8) commercial and industrial hazardous waste incinerators. The Agency's purpose was to gather accurate data on full-scale incinerator environmental performance, for the EPA's use in developing a regulatory impact analysis document in the near future for all such incinerators subject to existing RCRA standards. Because the performance information contained herein is of high interest to the hazardous waste incineration technical community in general, EPA wishes to make it available as a technical report at this time.

The material contained in this report has been reviewed and approved by experts within the Agency with state-of-the-art knowledge of the operation, testing, and evaluation of incinerator performance. During sampling and analysis of the incinerators in the field, EPA's most modern and scientific methods were employed along with the appropriate level of quality assurance and quality control. The incinerators were studied during a closely monitored mode of operation while disposing of mixtures of real-world hazardous wastes typical of their usual feedstocks. We are pleased to report that under these circumstances the incinerators were generally successful in performing their task of safely destroying hazardous waste and controlling other harmful emissions to the environment with some exceptions.

Requests for further information regarding this report on performance of full-scale incineration facilities should be directed to the Energy Pollution Control Division, IERL, Cincinnati.

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ABSTRACT

This report describes a study to evaluate current baseline performance of hazardous waste incinerators. Tests were conducted at eight full scale facilities to characterize all feed and effluent streams. Pollutants measured included EPA Appendix VIII hazardous compounds, particulates, hydrogen chloride, carbon monoxide, and total hydrocarbons. The data were analyzed to address questions on Destruction and Removal Efficiency (DRE), products of incomplete combustion (PIC), pollutant emission levels, performance of air pollution control systems, and relationships of process parameters to destruction efficiency and emission levels. A large base of data on performance of hazardous waste incinerators was accumulated, and many possible relationships between parameters were explored. Conclusions included: DREs were generally above 99.99%; measured DREs tended to be higher when concentration of the hazardous compound in the waste was higher; the most frequently observed PICs were benzene, toluene, chloroform, tetrachloroethylene, and naphthalene; the particulate EPA regulation of 180 mg/Nm³ was not routinely met; and the hydrogen chloride EPA regulation almost always met.

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The cooperation of all test sites and the efforts of many individuals in Midwest Research Institute's Environmental Systems and Analytical Chemistry Departments are also acknowledged.

APPENDIX C

SUMMARY OF RESULTS FROM TESTING AT
THE ROSS INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration regulations. Emissions data for the RIA are being provided by a sampling and analysis program. EPA selected the hazardous waste incinerator at Ross Incineration Services, Inc., as one facility for emission measurements.

A test was conducted at this site on June 10 and 11, 1982. Samples of waste feeds and gaseous, liquid, and solid effluents were collected and analyzed. The following sections of this appendix present a summary of destruction and removal efficiency (DRE) and stack effluent results, a brief process description, a synopsis of the test plan and sampling and analysis methods, a discussion of the test results, and a QA/QC discussion.

SECTION 2

SUMMARY OF DRE AND STACK EFFLUENT RESULTS

The DRE results for principal organic hazardous constituents (POHCs) for the test at Ross Incineration Services are summarized in Table C-1. Chloride emissions and removal efficiency, particulate emissions, and metals emissions are presented in Table C-2. These and other results are presented in more detail in Section 5.

DREs for five volatile and nine semivolatile POHCs were all above the 99.99% requirement in EPA's regulations for hazardous waste incinerators. Two additional volatile POHCs, carbon tetrachloride (CCl_4) and trichloroethylene (TCE) had DREs above or below 99.99%, depending on the sampling method used. Two methods were used to collect stack samples of the volatile POHCs, a volatile organic sampling train (VOST) and gas bags. (These methods are described in Section 4.) DREs calculated from emissions measured by the VOST were above 99.99% while DREs calculated from emissions measured by the gas bag method were below 99.99%.

The average hydrogen chloride (HCl) emissions and particulate emissions were below the requirements in EPA's regulations for hazardous waste incinerators. The HCl removal efficiency exceeded the EPA requirements. The average emission rates for lead and chromium, the two metals emitted in the greatest quantities, are shown on Table C-2. Data on other metals emitted in lesser quantities are presented in Section 5.

TABLE C-1. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs) FOR THE ROSS INCINERATOR (BLANK CORRECTED)

Volatile compounds	Gas bags			Fast VOST		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Carbon tetrachloride	99.9930	99.970	99.963	> 99.9964	> 99.9961	> 99.9959
Trichloroethylene	99.989	99.935	99.947	> 99.99936	99.9965	99.9969
Tetrachloroethylene	99.99925	99.99910	99.99951	> 99.9986	> 99.9977	99.99912
Toluene	99.99946	99.9987	99.9969	> 99.99904	> 99.9987	> 99.9978
Methylene chloride ^a	99.9974	99.82	99.72	> 99.968	> 99.989	> 99.978
Methyl ethyl ketone	99.99943	99.99918	99.99952	99.99967	99.99930	99.99932
1,1,1-Trichloroethane	> 99.99971	99.9979	99.9951	99.99952	> 99.9990	> 99.999
1,1,2-Trichloroethane	> 99.9999	> 99.9999	> 99.9999	> 99.999994	> 99.999994	> 99.999994
Semivolatile compounds						
NM5						
N,N-Dimethyl acetamide ^b	Run 1	Run 2	Run 3			
	> 99.9998	> 99.9999	> 99.9999			
	> 99.997	> 99.993	> 99.992			
	99.9992	99.9990	> 99.9994			
	> 99.994	> 99.994	> 99.991			
	> 99.9996	> 99.998	> 99.999			
	^c	> 99.99	> 99.99			
	> 99.998	> 99.998	> 99.998			
	> 99.998	> 99.998	> 99.998			
	> 99.9993	> 99.999	> 99.9991			

^a The methylene chloride results should be viewed with caution because of high blank values and the large difference in results between runs.

^b Dimethyl acetamide is not a POHC but was of interest to EPA.

^c Waste feed concentration < 100 µg/g in this run.

TABLE C-2. AVERAGE STACK EMISSIONS

DREs, %	VOST
<u>Hydrogen chloride emissions,</u> <u>g/min (lb/hr)</u>	1.86 (0.246)
<u>Hydrogen chloride removal</u> <u>efficiency, %</u>	99.940
<u>Particulate emissions, mg/dscm</u> <u>(gr/dscf) corrected to 7% O₂</u>	151 (0.0660)
<u>Metals emissions, g/min (lb/hr)</u>	
Lead	6.9 (0.91)
Chromium	0.19 (0.025)

SECTION 3

PROCESS DESCRIPTION AND PROCESS DATA

The incinerator is a commercial facility located in Ohio. It consists of a large, nearly rectangular combustion chamber and a rotary kiln as shown schematically in Figure C-1.

There are four waste feed streams to the incinerator. Aqueous waste is fed continuously into the combustion chamber above the rotary kiln exit into the chamber. Liquid organic waste is fed continuously at the opposite end, below the gas exhaust duct that is near the top of that end of the chamber. The other two waste feeds are both drummed waste. Depending on the waste characteristics, some drums are sliced into sections and conveyed into the kiln. Other drums are placed in groups of four on metal "sleds" and are conveyed through the combustion chamber, remaining in the chamber for about 4 hr. Normally, these latter drums are recycled back through the kiln after they have passed through the combustion chamber. This was not done during the tests because the test period was not long enough to encompass travel of drums through the combustion chamber and recycling them back through the kiln. The number of drums per hour fed to the chamber and kiln were four and an average of seven, respectively. No auxiliary fuel is used at this plant.

Gases exiting the combustion chamber pass through a water quench section and two packed scrubbers with caustic addition to adjust pH. The gases then pass through a two-stage ionizing wet scrubber (IWS) and into the 200 hp induced draft fan that discharges into the stainless steel stack. All liquid effluents are recycled to the two ponds, with makeup provided by nearby lake water. Thus, the only effluent from the incinerator, other than the stack, is the ash discharged from the rotary kiln.

The incinerator and associated scrubbers operated normally during the tests. Process data obtained during the tests are summarized in Table C-3, along with information needed for dispersion modeling. These data show that the combustion chamber temperature ranged from 1117 to 1154°C (2043 to 2110°F) and the calculated residence time was 6.2 to 6.7 sec. Average heat input rate to the incinerator, from all wastes, was 74×10^6 Btu/hr. Waste feed rates monitored during the tests are summarized in Table C-4.

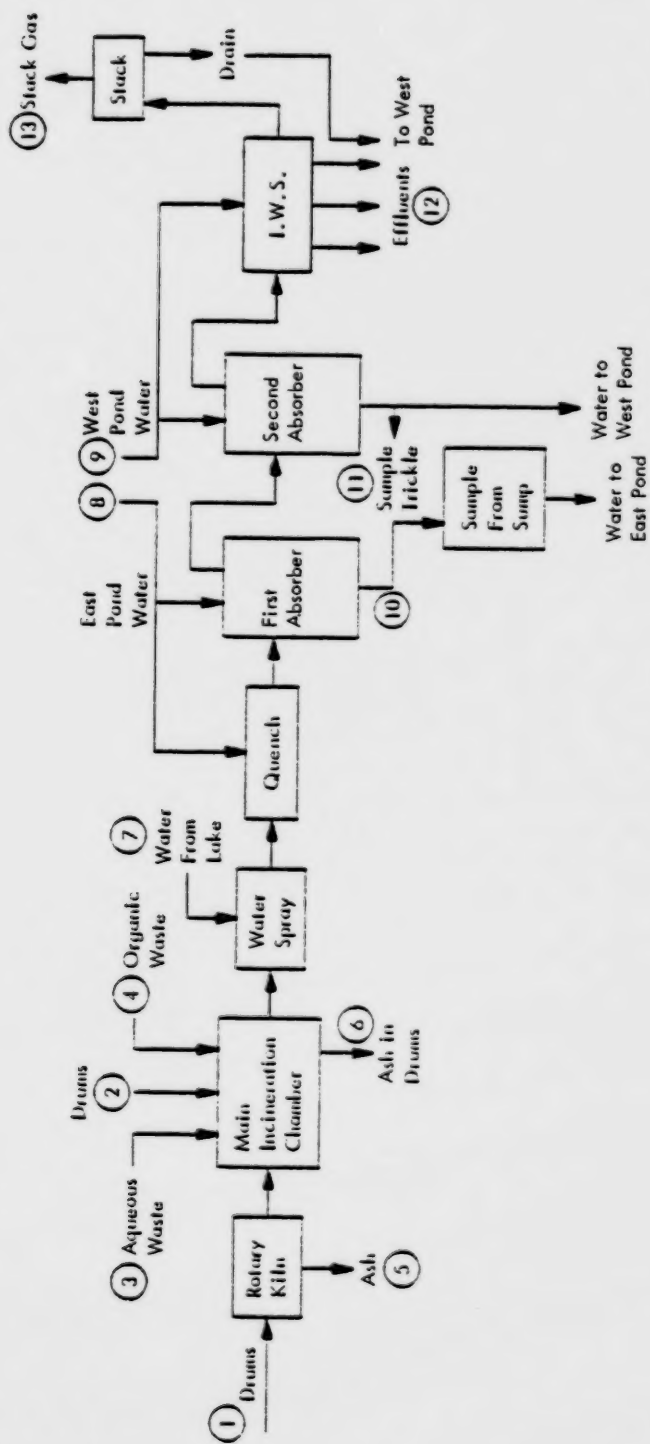


Figure C-1. Process diagram and sampling locations.

TABLE C-3. PROCESS DATA^a

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82	Average
Calculated residence time ^b (sec)	6.2	6.5	6.7	6.5
Combustion chamber temp., °C (°F)	1,154 (2,110)	1,146 (2,094)	1,117 (2,043)	1,139 (2,082)
Temp. exit dry quench, °C (°F)	132 (270)	132 (270)	132 (270)	132 (270)
Temp. into 1st absorber, °C (°F)	71 (160)	74 (166)	71 (160)	72 (162)
Heat input, kJ/hr (Btu/hr)	87 x 10 ⁶ (83 x 10 ⁶)	60 x 10 ⁶ (57 x 10 ⁶)	87 x 10 ⁶ (83 x 10 ⁶)	78 x 10 ⁶ (74 x 10 ⁶)
pH of 1st absorber effluent	7.75	7.75	7.90	7.80
Ionizing wet scrubber				
Section No. 1				
AC volts	224	222	235	227
AC amps	1	1	1	1
DC kilovolts	32	33	33	33
DC milliamps	14	13	15	14
Sparks/min	62	64	59	62
Section No. 2				
AC volts	314	316	385	338
AC amps	3	3	5	4
DC kilovolts	30	31	31	31
DC milliamps	36	33	42	37
Sparks/min	46	48	54	49
Stack height (m)	23	23	23	23
Stack exit velocity (m/min)	569	564	550	561
Stack temperature (°C)	56	59	58	58

^a Average of readings taken during each run.

^b Residence time computed from chamber volume, dry gas flow and temperature, and estimated moisture content of 10%.

TABLE C-4. WASTE FEED RATES

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Waste feed rates, kg/min (lb/hr)			
Drum waste to kiln	10.9 (1,440)	13.7 (1,810)	11.0 (1,450)
Drum waste to chamber	13.6 (1,800)	11.3 (1,490)	15.9 (2,100)
Aqueous waste	35.2 (4,650)	36.9 (4,870)	36.1 (4,770)
Organic waste	40.3 (5,320)	36.0 (4,750)	35.4 (4,670)

SECTION 4

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

Samples collected and subsequent analyses for this test are summarized in Table C-5, for each sampling point shown previously in Figure C-1.

Sampling of the drum feed to the incinerator required considerable effort. A total of about 200 drums each containing seven different types of waste had to be sampled. The chamber drums contained four different types of waste and the kiln drums contained three different types.

The plant set aside drums, previously selected in concert with EPA, for use in the tests. Because of the large number of drums, they were sampled on the day preceeding the tests. All drums were separated by type of waste. For each waste type, samples were transferred from groups of three drums into a bottle and the bottle labeled with the specific numbers for those three drums. A record was kept during each test of the number of every drum that entered the incinerator. After the test, the respective samples were used to prepare a composite sample of each type of waste used during that specific test (i.e., seven composite drum feed samples per test). Due to the nature of each type of drum waste, the composite of each type was analyzed separately.

Sampling of the material in the drums was difficult, due to the varying consistency and often heterogeneous nature of the material. In a few cases (about 20 drums), it was impossible to get the plug open on the top of the drum or to remove the lid. In several cases, the nature of the material made it impossible to sample in a way that one could be confident the sample was representative of that drum. However, a large number of samples were taken from many drums that contained essentially the same type of waste. It is believed that the composite samples are as representative as any that could have reasonably been obtained.

Aqueous and organic wastes fed continuously to the incinerator were sampled every 15 min during the tests to obtain a composite sample of each. A 50/50 mixture of CCl_4 and TCE was continuously pumped (spiked) into the organic waste feed line, at a rate of about 200 mL/min. The sampling point for the organic waste feed was about 8 ft downstream of the valve through which the spiking mixture was pumped into the waste feed line.

Sampling of other effluents consisted of grab sampling, except for the stack. Stack sampling consisted of Modified Method 5 (for particulates, Cl⁻ metals and semi-volatile POHCs); two methods for sampling volatile POHCs, gas bags and a volatile organic sampling train; and continuous monitors for

TABLE C-5. SUMMARY OF SAMPLING AND ANALYSIS ACTIVITY

Sampling point	No. of samples per run	Sampling technique	Analysis
<u>Waste feeds</u>			
1. Drums into rotary kiln	1 Composite	1 Sample per drum	HHV, ash POHCs, ultimate Cl H ₂ O 12 Metals
2. Drums into main chamber	1 Composite	1 Sample from each drum present in the inciner- ator during the test time	As above
3. Aqueous waste	1 Composite	Collect every 15 min	As above
4. Organic waste	1 Composite	Collect every 15 min	As above, and viscosity
<u>Ash</u>			
5. Rotary kiln ash	1 Composite	3 Grab samples	Archive
6. Main chamber ash	1 Composite	1 Grab per hour (i.e., for each drum sled)	Semi-volatile POHCs Archive
<u>Scrubber liquids</u>			
7. Water spray (lake water)	1 Grab	1 Grab	Archive
8. East pond water to quench and first scrubber	1 Grab	1 Grab	POHCs Cl
(continued)			

TABLE C-5. (continued)

Sampling point	No. of samples per run	Sampling technique	Analysis
<u>Scrubber liquids (continued)</u>			
9. West pond water to second scrubber and ionized wet scrubber	1 Grab	1 Grab	POHCs Archive
10. Effluent from absorber unit and quench	1 Composite	3 Grabs	pH POHCs Cl 12 Trace metals
11. Effluent from second absorber	1 Composite	3 Grabs	Semi-volatile POHCs Archive
12. Ionized wet scrubber effluents and stack drain	1 Composite	3 Grabs for each of four effluents	Semi-volatile POHCs Archive
<u>Stack</u>			
13. MM5 Train	1	MM5 Train	Particulate weight 12 Metals on particulate Semivolatile POHCs Semivolatile PICs Cl
Volatiles train	1	Tenax train and Tedlar gas bags	Volatile POHCs Volatile PICs
CO ₂ and O ₂	1	Orsat	CO ₂ and O ₂
CO, CO ₂ , O ₂ , and HC	Continuous	Continuous monitors	CO, CO ₂ , O ₂ , HC

O₂, CO₂, CO, and total hydrocarbons. A schematic diagram of the MM5 train is shown in Figure C-2 and a diagram of the VOST train is shown in Figure C-3.

Gas bag samples were collected in 15-L Tedlar bags, drawing gas into the bags over a period of about 1 hr. The VOST train consists of a pair of Tenax and Tenax/charcoal traps. Gas is drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The pair of traps is then removed and replaced with another pair, until a total of six pairs have been used (i.e., 120 min of sampling time). These traps and the gas bags are then returned to the analytical laboratory for analysis of volatile POHCs.

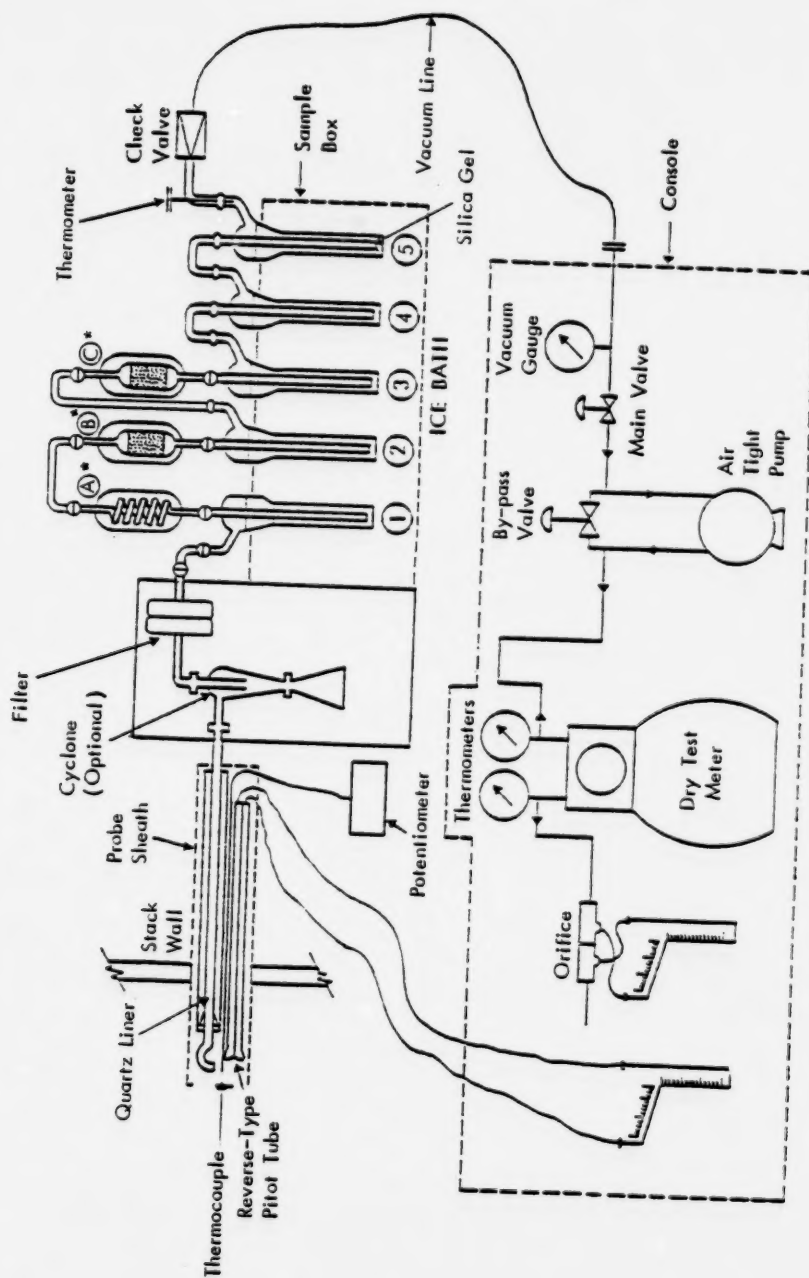
POHCs in the waste feed and stack samples are analyzed by GC/MS. The waste feed samples are first homogenized using either manual or mechanical homogenization. Liquid waste feeds can generally be homogenized manually while drummed wastes often require a mechanical mixer.

The homogenized feed samples are analyzed for volatile POHCs using a method based on Method A101b in A.D. Little report "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0-g aliquot of the mixed waste feed sample is dispersed in 40 mL of prepurified tetraglyme. A 10- μ L aliquot of the tetraglyme solution is spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample is then analyzed by the purge-trap-desorb technique as described in A. D. Little Method A101A (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis may be repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs are analyzed in the waste feed samples according to A. D. Little Method P023. A 5-g portion of the homogenized waste feed sample is spiked with semivolatile surrogate compounds, such as decafluorobiphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 μ g/g depending on the expected sample complexity. The sample is allowed to equilibrate with the spiked compounds and then is extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appears on the surface of the methylene chloride, the pH is checked and it is neutralized. The extract is then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

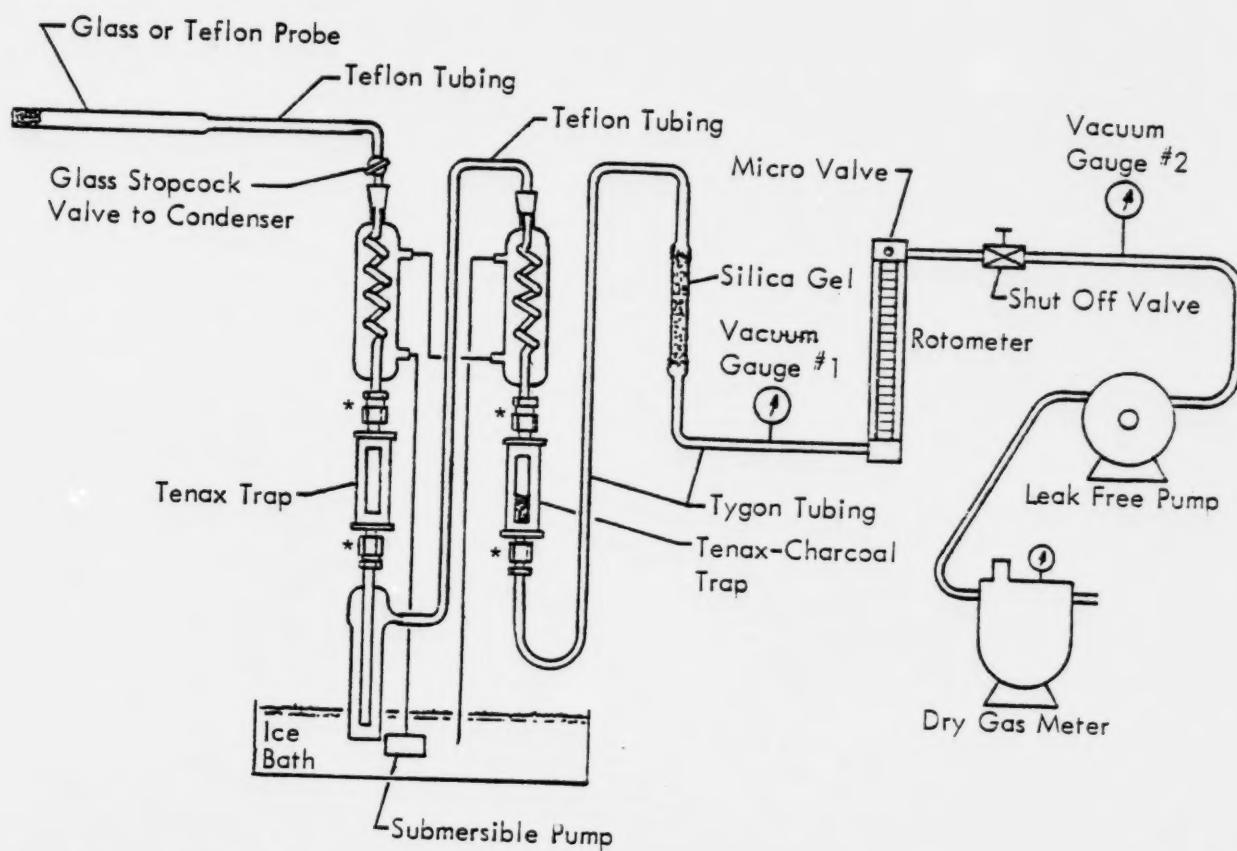
An aliquot of the extract is then screened by gas chromatography with a flame ionization detector. If necessary, the extract is further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column (FSCC). A 1.0-mL aliquot of the extract is then spiked with 20 μ g of D₁₀-anthracene internal standard and analyzed by FSCC GC/MS.

The volatile POHCs from the stack gas samples are contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with the VOST. A known amount of the gas bag sample, usually 5.0 L, is transferred onto a



- ① Modified Greenburg-Smith, Reversed, Empty
 - ② Greenburg-Smith, 50 ml of Double Distilled in Glass H_2O
 - ③ Greenburg-Smith, 100 ml of 0.1N KOH
 - ④ Modified Greenburg-Smith, Empty
 - ⑤ Modified Greenburg-Smith, SiO_2
- (A) Condenser
 - (B) XAD Resin Cartridge #1
 - (C) XAD Resin Cartridge #2
 - * Ice Water Jacket

Figure C-2. Modified Method 5 train (MM5).



* Special O-Ringed Stainless Steel Fittings

Figure C-3. Volatile organic sampling train (VOST).

Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). A needle is inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps are analyzed using the same techniques as for the VOST traps described below.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards are spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene (BFB) internal standards using the flash vaporization technique in which the spiking solution is vaporized and carried onto the trap with a carrier gas. The glass traps are attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, is turned on through the trap and a syringe needle injected through the GC septum port. One µL of a 25-ng/µL methanolic solution of the internal standards is slowly expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap is shut off and the syringe removed. The total flow of gas through the traps during spiking is about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) are thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL) which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps are desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds pass into the bottom of the water column, are purged from the water, and then are collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds are then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs in the stack samples involves several components of the MM5 train. Each of the components of the MM5 train are spiked with a separate surrogate standard according to the following scheme:

Front XAD-2 resin trap: 20 µg of ¹³C-tetrachlorobiphenyl
Back XAD-2 resin trap: 20 µg of decafluorobiphenyl
Particulate filter plus probe rinse residue: 20 µg of D₁₂-chrysene
Condensate water: 20 µg of D₈-naphthalene

The spiked components are normally extracted separately, and then the extracts are combined, dried with anhydrous sodium sulfate, concentrated to 1.0 mL using Kuderna-Danish evaporation according to A. D. Little Method P031 and using supplementary gentle nitrogen blowdown, and analyzed by FSCC GC/MS. The samples are extracted according to the following procedures:

XAD-2 resin traps: The XAD-2 resin traps are Soxhlet-extracted with methylene chloride for 16 hr as described in A. D. Little Method P024b.

Particulate filters plus probe rinse residues: The residue from the acetone rinse of the probe is added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in A. D. Little Method P024b.

Condensate water: The condensate water is extracted with three 60-mL aliquots of methylene chloride as described in A. D. Little Method P021a (equivalent to EPA Method 625).

Analysis for POHCs in scrubber water and ash samples is similar to the procedures described above. Metals analysis is by acid digestion of samples and analysis by inductively coupled argon plasma emission spectrophotometry and/or atomic absorption spectrophotometry. Particulate and chloride analyses are conducted according to EPA Method 5 and EPA Method 325.2 from "Methods for Chemical Analysis of Water and Wastes," respectively. More detailed descriptions of all the above sampling and analysis methods are presented in Appendix B.

SECTION 5

DISCUSSION OF RESULTS

DESTRUCTION/REMOVAL EFFICIENCY FOR POHCs

DREs were determined for all POHCs listed by EPA which were found to be present in the waste feeds. No attempt was made to restrict the number of POHCs based on any selection or ranking method. Since prior information was available about some POHCs likely to be present in the waste, those POHCs were quantified against authentic standards. Others found to be present in the waste were semi-quantified from the GC/MS analysis based on total ionization of each compound relative to an internal standard spiked into the samples (D_{10} -anthracene for semi-volatile POHCs and P_4 -1,2-dichloroethane for volatile POHCs).

DREs were calculated from the input rate of each POHC in the waste feeds, and the output rate in the stack effluent. Feed rates for each of the waste feeds were presented earlier in Table C-4. Concentrations of POHCs identified in those feeds are given in Table C-6, and the calculated input rate for each POHC is shown in Table C-7. Output concentration of the volatile POHCs in the stack effluent are shown in Table C-8. Emission rates for volatile POHCs are presented in Table C-9. Semivolatile emissions are presented in Table C-10. These values were calculated from the stack flow rate (Method 5) and from analysis of the components of the Modified Method 5 train for semi-volatile POHCs and analysis of the gas bag and VOST samples for volatile POHCs.

The DREs calculated were presented in Table C-1. All the DREs equalled or exceeded 99.99%, except for CCl_4 and TCE in Runs 2 and 3, as determined from the gas bags. However, results from the VOST method for CCl_4 and TCE exceeded 99.99%. None of the results indicate that the concentrations determined with the VOST trap samples are erroneously low, nor do they give any strong basis for suspecting that the bag samples gave erroneously high results. Consequently, it is not possible to resolve the difference between the gas bag and VOST sample results, or to ascertain which DRE, for CCl_4 and TCE, is more likely the correct one.

EMISSION OF PICs

Analysis of samples included identification of PICs, i.e., those compounds identified in the stack samples that are listed as POHCs but which were not found in any of the waste feed samples at concentrations above 100 $\mu\text{g/g}$. The concentration and output rate for volatile compounds are tabulated in Tables C-11 and C-12, respectively. Semivolatile emissions are found in Table C-13.

TABLE C-6. CONCENTRATION OF PHHCs IN WASTE FEED (µg/g)

	Concentration of PHHCs in drum waste to chamber (µg/g)			Concentration of PHHCs drum waste to kiln (µg/g)			Concentration of PHHCs in aqueous waste (µg/g)			Concentration of PHHCs in organic waste (µg/g)		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Volatile PHHCs^a												
Carbon tetrachloride	ND	ND	ND	ND	ND	ND	ND	ND	ND	4,100	5,900	5,700
Trichloroethylene	40,000	15,000	34,000	370	44	7	ND	ND	ND	12,000	8,100	7,800
Tetrachloroethylene	30,000	35,000	85,000	120	73	41	ND	ND	ND	9,300	7,900	8,100
Toluene	70,000	61,000	57,000	28,000	7,600	8,600	33	24	25	69,000	56,000	48,000
Volatile PHHCs^b												
Methylene chloride	17,000	58,000	22,000	ND	ND	ND	42	90	27	ND	ND	ND
Methyl ethyl ketone	7,400	6,600	14,000	2,100	2,200	2,800	290	360	330	18,000	18,000	38,000
1,1,1-Trichloroethane	4,000	2,200	400	ND	ND	ND	ND	ND	ND	62,000	24,000	16,000
1,1,2-Trichloroethane	2,600	2,400	2,300	ND	ND	ND	ND	ND	ND	ND	ND	ND
Semi-volatile PHHCs^a												
N,N-Dimethylacetamide ^c	ND	ND	ND	76,000	130,000	170,000	180	230	220	ND	ND	ND
Phenol	ND	ND	ND	660	ND	ND	130	160	140	ND	ND	ND
2,4-Dimethylphenol	ND	ND	ND	ND	ND	ND	ND	ND	ND	600	500	2,000
Naphthalene	520	720	470	160	790	ND	ND	ND	ND	570	450	490
Semi-volatile PHHCs^b												
Butyl benzyl phthalate	6,800	820	1,400	ND	ND	ND	ND	ND	ND	210	160	140
Phthalic anhydride	ND	440	160	ND	ND	220	17	71	47	ND	ND	ND
Aniline	ND	ND	ND	2,400	1,500	2,400	ND	ND	ND	ND	ND	ND
Methyl pyridine (picoline)	ND	ND	ND	2,300	3,000	3,600	ND	ND	ND	ND	ND	ND
Cresol(s)	ND	ND	ND	ND	ND	ND	20	20	10	2,900	2,000	2,500

ND = None detected. Compounds present at < 100 µg/g are not shown as PHHCs.

^a Quantitated against authentic standard.^b Semiquantitated based on total ionization relative to internal standard.^c Dimethylacetamide is not on the PHHC list but was of interest to EPA.

TABLE C-7. CALCULATED INPUT RATE FOR POHCs IN WASTE FEEDS

	Input rate (g/min)		
	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
<u>Volatile POHCs^a</u>			
Carbon tetrachloride	160	210	200
Trichloroethylene	1,040	460	820
Tetrachloroethylene	780	680	1,640
Toluene	4,040	2,810	2,700
<u>Volatile POHCs^b</u>			
Methylene chloride	230	660	350
Methyl ethyl ketone	860	770	1,610
1,1,1-Trichloroethane	2,550	890	570
1,1,2-Trichloroethane	35	27	37
<u>Semivolatile POHCs^a</u>			
N,N-Dimethylacetamide ^c	830	1,780	1,870
Phenol	12	6	5
2,4-Dimethylphenol	20	20	70
Naphthalene	32	35	24
<u>Semivolatile POHCs^b</u>			
Butyl benzyl phthalate	100	17	27
Phthalic anhydride	d	8	7
Aniline	26	21	26
Methyl pyridine	25	41	40
Cresol(s)	120	73	90
Total	10,860	8,508	10,086

^a Quantitated against authentic standard.

^b Semiquantitated based on total ionization relative to internal standard.

^c Dimethylacetamide is not on the POHC list but was of interest to EPA.

^d Concentration less than 100 µg/g in any waste.

TABLE C-8. CONCENTRATION OF VOLATILE POHCs IN STACK EFFLUENT (NOT BLANK CORRECTED), ng/L

	Gas bags			Run 1			VOST		
	Run 1	Run 2	Run 3	3rd Pair	4th Pair	2nd Pair	Run 2	3rd Pair	4th Pair
Carbon tetrachloride ^a	14	80	96	3.6	13	4.5	5.5	19	5.7
Trichloroethylene ^a	140	380	560	3.7	15	17	9.6	59	12
Tetrachloroethylene ^a	8.0	8.6	11	1.1	27	2.7	2.0	36	4.3
Toluene ^a	32	52	110	10	82	19	18	106	56
Methylene chloride ^{b,c}	11	1,500	1,200	89	d	27	110	25	17
Methyl ethyl ketone ^b	0.6	0.8	1.0	3.6	3.8	7.0	9.2	14	15
1,1,1,-Trichloroethane ^b	9	52	64	25	14	11	19	7.9	7.3
1,1,2-Trichloroethane ^b	< 0.2	< 0.2	< 0.2	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05	< 0.05

^a Quantified against authentic standard.

^b Semiquantified based on total ionization relative to internal standard.

^c The methylene chloride results should be viewed with caution because of high blank values and the large difference in results between runs.

^d Data missed during GC/MS acquisition.

TABLE C-9. EMISSION RATE OF VOLATILE POHCs IN STACK EFFLUENT (NOT BLANK CORRECTED), g/min

[illegible]^a Quantified against internal standard.

^b Semiquantified based on total ionization relative to internal standard.

^c Data missed during GC/MS acquisition.

TABLE C-10. STACK EMISSIONS FOR SEMIVOLATILE POHCs

	Concentration (ng/L)		
	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
N,N-Dimethylacetamide ^{a,c}	< 2	< 2	< 2
Phenol ^a	< 0.4	< 0.5	< 0.5
2,4-Dimethylphenol ^a	0.24	0.25	< 0.5
Naphthalene ^a	< 2	< 2	< 2
Butyl benzyl phthalate ^b	< 0.4	< 0.5	< 0.5
Phthalic anhydride ^b	< 0.8 ^d	< 0.8	< 0.9
Aniline ^b	< 0.4	< 0.5	< 0.5
Methyl pyridine ^b	< 0.4	< 0.5	< 0.5
Cresol(s) ^b	< 0.8	< 0.8	< 0.9

	Output rate (g/min)		
	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
N,N-Dimethylacetamide ^{a,c}	< 0.002	< 0.002	< 0.002
Phenol ^a	< 0.0004	< 0.0004	< 0.0004
2,4-Dimethylphenol ^a	0.0002	0.0002	< 0.0004
Naphthalene ^a	< 0.002	< 0.002	< 0.002
Butyl benzyl phthalate ^b	< 0.0004	< 0.0004	< 0.0004
Phthalic anhydride ^b	< 0.0008 ^d	< 0.0007	< 0.0008
Aniline ^b	< 0.0004	< 0.0004	< 0.0004
Methyl pyridine ^b	< 0.0004	< 0.0004	< 0.0004
Cresol(s) ^b	< 0.0008	< 0.0007	< 0.0008

^a Quantitated against authentic standard.

^b Semiquantitated based on total ionization relative to internal standard.

^c Dimethylacetamide is not on the POHC list but was of interest to EPA.

^d Concentration of phthalic anhydride was < 100 µg/g in waste for Run 1.

TABLE C-11. CONCENTRATION OF VOLATILE PICs IN STACK EFFLUENT (NOT BLANK CORRECTED), ng/L

	Gas bags				Run 1				VOST			
	Run 1	Run 2	Run 3		3rd Pair	4th Pair	2nd Pair		Run 2	3rd Pair	4th Pair	Run 3
Chloroform ^a	7.8	9.6	9.6		9.6	10.1	7.9		14	5.4	8.3	9.1
Benzene ^a	11	20	24		7.8	7.4	15		20	10	11	7.9
Bromomethane ^a	7.4	1.2	0.8		0.3	c	2.9		1.8	1.2	1.6	1.5
Chloromethane ^b	220	48	76		4.1	c	6.0		9.8	6.8	1.8	2.5
Carbon disulfide ^b	26	3.5	4.4		44	c	25		58	18	41	15
Bromochloromethane ^b	11	38	50		19	c	15		30	16	17	24
Methylene bromide ^b	9.2	12	18		11	11	20		22	20	19	33
Bromodichloromethane ^b	4.8	7.0	3.6		4.2	6.4	5.5		5.9	4.8	4.9	8.4
Dibromochloromethane ^b	2.6	1.5	3.0		1.0	4.7	6.0		3.0	6.3	6.0	8.9
Bromoform ^b	6.2	4.6	6.6		0.4	8.6	11		12	11	14	16

^a Quantified against authentic standard.

^b Semiquantified based on total ionization relative to internal standard.

^c Data missed during GC/MS acquisition.

TABLE C-12. EMISSION RATE OF VOLATILE PICs IN STACK EFFLUENT (NOT BLANK CORRECTED), g/min

Compound	Gas bags				VOST			
	Run 1		Run 2		Run 1		Run 2	
	Run 1	Run 2	Run 3	Run 4	3rd Pair	4th Pair	2nd Pair	3rd Pair
Chloroform ^a	0.0064	0.0076	0.0074	0.0078	0.0082	0.0062	0.011	0.0065
Benzene ^a	0.0090	0.016	0.019	0.0064	0.0060	0.012	0.016	0.0087
Bromomethane ^b	0.0060	0.00094	0.00062	0.00024	c	0.0023	0.0014	0.0013
Chloromethane ^b	0.18	0.038	0.059	0.0033	c	0.0047	0.0077	0.0014
Carbon disulfide ^b	0.021	0.0028	0.0034	0.036	c	0.020	0.046	0.032
Bromochloromethane ^b	0.0090	0.030	0.039	0.016	c	0.012	0.024	0.013
Methylene bromide ^b	0.0075	0.0095	0.014	0.0090	0.0090	0.016	0.017	0.015
Bromodichloromethane ^b	0.0039	0.0055	0.0028	0.0034	0.0052	0.0043	0.0046	0.0039
Dibromochloromethane ^b	0.0021	0.0012	0.0023	0.00082	0.0038	0.0047	0.0024	0.0047
Bromoform ^b	0.0050	0.0036	0.0051	0.00033	0.0070	0.0087	0.0095	0.011
								0.0085
								0.012

^a Quantified against authentic standard.

^b Semiquantified based on total ionization relative to internal standard.

^c Data missed during GS/HS acquisition.

TABLE C-13. STACK EMISSIONS FOR SEMIVOLATILE PICs
(NOT BLANK CORRECTED)

	Concentration (ng/L)		
	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Fluoranthene ^a	1.5	1.3	1.3
Pyrene ^a	1.3	< 0.5	1.3
	Output rate (g/min)		
	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Fluoranthene ^a	0.0012	0.001	0.001
Pyrene ^a	0.0011	< 0.0004	0.001

^a Semiquantitated based on total ionization relative to internal standard.

TABLE C-14. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>				
Carbon tetrachloride	2	a	44	50
Trichloroethylene	18		32	33
Tetrachloroethylene	4		14	16
Toluene	25		620	720
Methylene chloride	19		3,600	390
Methyl ethyl ketone	0		3.5	0.7
1,1,1-Trichloroethane	140		86	47
1,1,2-Trichloroethane	< 0.2		< 1	0
<u>PICs</u>				
Chloroform	2	a	52	11
Benzene	6		130	16
Bromomethane	< 1		< 1	0
Chloromethane	49		8.4	0.5
Carbon disulfide	8		9.5	13
Bromochloromethane	< 1		< 1	0
Methylene bromide	< 1		< 1	0
Bromodichloromethane	< 1		< 1	0
Dibromochloromethane	< 1		< 1	0
Bromoform	< 1		< 1	0

^a Only one gas bag blank was used.

BLANK VALUES FOR POHCs AND PICs

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table C-14. The sample volumes for gas bags and VOST are presented in Table C-15. Blank correction data for semivolatile POHCs and PICs are presented in Table C-16. Sample volumes for the MM5 train can be found in Table C-19.

DRE VALUES FOR VOLATILES AND SEMIVOLATILES

Destruction and removal efficiencies (DREs) based on the above data were presented in Table C-1. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$\text{DRE} = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

POHCs AND Cl^- IN SCRUBBER WATER STREAMS

The test included sampling and analysis of the influent/effluent from the first absorber in the incinerator process, since it would be expected that this would be the scrubber most likely to provide any removal of POHCs present in the gas from the combustion chamber. Results given in Table C-17 show that only one compound (dimethylacetamide) was above the detection limits. Because of the presence of dimethylacetamide in the first absorber effluent, samples of the second absorber and IWS water streams were later analyzed for semi-volatile organics. Those data are also presented in Table C-17.

Table C-17 also presents Cl^- concentrations for the influent/effluent of the first absorber.

POHCs IN ASH

One sample of combustion chamber ash collected during Run 2 was analyzed for semi-volatile POHCs. The only POHC detected was naphthalene at a concentration of 67 $\mu\text{g/g}$ of ash.

PARTICULATE EMISSIONS

The particulate emission data are presented in Table C-18. Associated Method 5 test data, which are also necessary for calculation of DREs, are given in Table C-19. The particulate emission data show that results for all three tests were below the concentration of 180 mg/dscm (corrected to 7% O_2) specified in EPA's regulations.

CONTINUOUS MONITORING DATA

Continuous monitors were used to determine concentrations of O_2 , CO_2 , CO , and HC in the stack effluent throughout each test period. The results are summarized in Table C-20. The concentrations of HC and CO were both low; 0 to 2.3 ppm for HC and 2.9 to 13.5 ppm for CO .

TABLE C-15. VOLATILE SAMPLE VOLUMES (DRY STANDARD LITERS)

Gas bags		VOST	
Run No.	Volume (L)	Run No./Pair No.	Volume (L)
1	5.0	1/3	18.9
2	5.0	1/4	18.6
3	5.0	2/2	18.2
		2/3	18.5
		2/4	18.2
		3/3	18.9
		3/4	19.0

TABLE C-16. SEMIVOLATILE BLANK CORRECTION VALUES

Compound	Blank correction value (μg)
<u>POHCs</u>	
N,N-Dimethyl acetamide	< 1
Phenol	4.0
2,4-Dimethyl phenol	< 1
Naphthalene	6.6
Butyl benzyl phthalate	< 1
Phthalic anhydride	< 2
Aniline	< 1
Methyl pyridine	< 1
Cresol(s)	< 2
<u>PICs</u>	
Fluoranthene	< 1
Pyrene	< 1

TABLE C-17. CONCENTRATION OF POHCs AND Cl^- IN SCRUBBER WATER STREAMS

	First absorber				Second absorber/IWS		Second absorber		IWS	
	Run 1, 6/10/82		Run 2, 6/11/82		Run 2, 6/11/82		Run 2, 6/11/82		Run 2, 6/11/82	
	Input	Effluent	Input	Effluent	Input	Effluent	Input	Effluent	Input	Effluent
Volatile POHCs ($\mu\text{g/L}$)										
Carbon tetrachloride	ND ^a	ND ^a	ND ^a	ND ^a	ND ^a	ND ^a	ND ^a	ND ^a	ND ^a	ND ^a
Trichloroethylene										
Tetrachloroethylene										
Toluene										
Methylene chloride										
Methyl ethyl ketone										
1,1,1-Trichloroethane										
1,1,2-Trichloroethane										
Semi-volatile POHCs ($\mu\text{g/L}$)										
N,N-Dimethylacetamide	140									
Phenol	< 1 ^b	56	< 1 ^b	270	19	220	119	92	0.6	ND ^a
2,4-Dimethylphenol	ND ^b	< 1 ^b	ND ^b	< 1 ^b	ND ^b	ND ^b	ND ^b	ND ^b	ND ^b	ND ^b
Naphthalene		180		170		170				
Butyl benzyl phthalate										
Phthalic anhydride										
Aniline										
Methyl pyridine										
Cresol(s)										
Cl^- (g/L) ^d	17.6	16.6	17.4	19.0	17.0	19.6				

^a ND = Nondetected; detection limit is 10 $\mu\text{g/L}$.

^b ND = Nondetected; detection limit is 1 $\mu\text{g/L}$.

^c Recovery of N,N-Dimethylacetamide from water is about 5% thus, actual concentrations may be 20 times the values presented.

^d Measurements by pH paper in the field showed (for all three runs) a pH of 7 for the effluent from first absorber and a pH of 5 for the effluent from second absorber.

TABLE C-18. PARTICULATE EMISSION DATA

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Particulate concentration, mg/dscm (gr/dscf)			
Measured	94.4 (0.0413)	117 (0.0510)	98.3 (0.0430)
Corrected to 7% O ₂ ^a	139 (0.0609)	176 (0.0770)	139 (0.0609)

^a Measured emissions were corrected to 7% O₂ as follows:

$$C_{\text{corr}} = \frac{(14\%)}{(21 - \% \text{ O}_2)} (C_{\text{measured}})$$

TABLE C-19. METHOD 5 TEST DATA

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Vol. of gas sampled (Nm ³)	2.101	2.155	1.988
Sampling time (min)	160	160	160
Percent isokinetic	102	108	102
Moisture content (%)	17	19	19
Percent O ₂ (dry)	11.5	11.7	11.1
Percent CO ₂ (dry)	7.1	6.9	7.3
Stack flow rate (act. m ³ /min)	1,144	1,133	1,106
Stack temperature (°C)	56	59	58
Stack flow rate (Nm ³ /min)	816	789	776

TABLE C-20. CONTINUOUS MONITORING DATA^a

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Oxygen (%)			
Range	7.8-12.1	6.9-12.8	7.6-12.5
Average	10.4	10.5	10.7
Carbon dioxide (%)			
Range	7.5-8.7	7.6-9.4	7.4-8.6
Average	7.9	8.3	8.1
Carbon monoxide (ppm _v)			
Range	2.1-7.4	4.7-15.5	2.9-8.7
Average	4.8	9.1	4.7
Total hydrocarbons (ppm _v) ^{b, c}			
Range	< 1	0.8-2.3	< 1-2.3
Average	< 1	0.9	1.0

^a Concentrations on dry gas basis.

^b Total HC reported as propane.

^c One ppm_v is approximately 1.6 g/min.

It is interesting to note that the total output rate for POHCs and PICs, as discussed earlier, was less than 1.6 g/min which is approximately equivalent to the 1 ppm detection limit of the continuous hydrocarbon analyzer.

ANALYSIS OF WASTE FEEDS

The waste feeds were analyzed for elemental content as well as heating value, ash, and viscosity. The results are presented in Table C-21.

HCl REMOVAL EFFICIENCY

HCl removal efficiency was calculated from measurements of the concentration of HCl in the stack effluent, the waste feed rates (Table C-4), and the Cl concentration in the waste feeds (Table C-21). The results are shown in Table C-22. HCl removal efficiency was above 99.9% for all runs, which is above the efficiency specified in EPA's regulations.

METALS ANALYSIS

Samples of waste feeds, first absorber effluent, and the stack particulate were analyzed for metals.

Table C-23 presents the concentrations of 12 metals in the waste feed samples for each of the three runs. Metals were present at relatively low concentrations in the drum waste to the kiln and the aqueous waste. The metal present at the highest concentration in the drum waste fed to the chamber was nickel. The liquid organic waste feed contained five metals at concentrations higher than 100 µg/g (barium, cadmium, chromium, antimony, and lead). Lead was present at the highest concentrations, ranging from 1,800 to 2,090 µg/g. Correspondingly, lead was the highest concentration metal in the effluent from the first absorber, as shown in Table C-24.

Metals found to be present at the highest concentrations in the particulate emissions, as shown in Table C-25, were the same as those in the organic waste feed. Perhaps of most significance was the relatively high concentration of lead in the particulate, which ranged from 68,900 to 96,100 µg/g, equivalent to 6.89 to 9.61% of the particulate. Based on the lead content of the waste feeds, and the particulate emission rate, it is estimated that 10% of the lead present in the wastes was emitted as part of the particulate emissions.

Table C-26 presents input and output mass rates for all metals that were measured above the detection limit for any sample.

QUALITY ASSURANCE SUMMARY

Quality assurance activities were divided into three categories: (1) external auditing; (2) sampling QC activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated so as to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

TABLE C-21. ANALYSES OF WASTE FEEDS

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82		
	Chlorine (%)				
Drum waste to kiln ^a	0.0193	1.60	0.12		
Drum waste to chamber ^b	7.63	7.46	6.68		
Aqueous waste	0.04	0.04	0.03		
Organic waste ^c	6.23	5.62	5.45		
	Water (%)				
Drum waste to kiln ^a	26.88	18.54	13.98		
Drum waste to chamber ^b	2.34	8.37	7.28		
Aqueous waste	97.68	94.14	94.62		
Organic waste	24.41	20.68	23.13		
	Heating value, kJ/kg (Btu/lb)				
Drum waste to kiln ^a	18,660 (8,037)	21,790 (9,383)	22,090 (9,512)		
Drum waste to chamber ^b	23,730 (10,220)	19,000 (8,180)	23,810 (10,253)		
Aqueous waste	1,560 (674)	1,560 (670)	1,510 (651)		
Organic waste	21,870 (9,420)	20,130 (8,670)	22,210 (9,564)		
	Ash (%)				
Drum waste to kiln ^a	36.94	39.98	40.97		
Drum waste to chamber ^b	0.55	0.51	0.25		
Aqueous waste	0.25	0.25	0.26		
Organic waste	2.42	2.12	2.28		
Ultimate analysis, Run 2	Percent C	Percent H	Percent N	Percent S	Percent O (by difference)
Drum waste to kiln ^a	32.01	6.29	0.007	0.038	20.17
Drum waste to chamber ^b	63.89	9.31	0.048	0.094	18.69
Aqueous waste	3.34	10.36	0.37	0.007	85.63
Organic waste	47.50	9.59	0.64	0.16	34.37

^a Weighted average based on feed rate and analyses of three types of waste.

^b Weighted average based on feed rate and analyses of four types of waste.

^c The organic waste in Run 2 had a viscosity of 298.6 sec at 100°F.

TABLE C-22. CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

	Run 1, 6/10/82	Run 2, 6/11/82	Run 3, 6/11/82
Cl ⁻ in feed, g/min (lb/hr)	3,565 (471)	3,280 (433)	2,790 (368)
Cl ⁻ out stack, g/min (lb/hr)	1.13 (0.149)	2.24 (0.296)	2.20 (0.290)
Cl removal efficiency (%)	99.968	99.932	99.921

TABLE C-23. CONCENTRATION OF METALS IN WASTE FEEDS (µg/g)

Method	Drum waste to chamber			Drum waste to kiln			Aqueous waste			Organic waste		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Silver	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.03	< 0.2	< 0.2	< 0.2	< 0.3	< 0.3	< 0.3
Arsenic	< 3.5	< 3.5	< 3.5	< 3.5	< 3.5	< 3.5	< 15	< 15	< 15	< 14	< 14	< 14
Barium	14.0	4.60	9.51	1.68	0.83	1.63	1.42	1.22	1.18	1,460	1,160	1,150
Beryllium	< 0.15	< 0.15	< 0.15	< 0.15	< 0.15	< 0.15	< 0.3	< 0.3	< 0.3	< 0.2	< 0.2	< 0.2
Cadmium	0.20	0.98	1.98	< 0.016	< 0.016	< 0.016	< 0.034	< 0.034	< 0.034	153	153	15
Chromium	50	442	123	13.0	1.43	2.38	0.179	0.063	< 0.034	542	431	425
Mercury	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 3	< 4	< 4	< 4
Nickel	476	3,100	703	42.2	9.08	8.81	0.092	0.081	< 0.063	94	26	27
Lead	25.2	25.2	25.7	0.92	< 0.25	0.31	4.04	2.98	2.33	2,090	1,830	1,800
Antimony	< 3	< 3	< 3	< 3	< 3	< 3	< 0.184	< 0.184	< 0.184	17	437	373
Selenium	< 20	< 20	< 20	< 20	< 20	< 20	< 2	< 2	< 2	< 21	< 21	< 21
Thallium	< 4	< 4	< 4	< 4	< 4	< 4	< 6	< 6	< 6	< 9	< 9	< 9

TABLE C-24. CONCENTRATION OF METALS IN FIRST
ABSORBER EFFLUENT ($\mu\text{g/L}$)

Metal	Run 1	Run 2	Run 3
Silver	< 200	< 200	< 200
Arsenic	< 15,000	< 15,000	< 15,000
Barium	2,580	1,920	1,470
Beryllium	< 300	< 300	< 300
Cadmium	1,280	1,270	1,170
Chromium	802	739	736
Mercury	< 3,000	< 3,000	< 3,000
Nickel	293	238	438
Lead	14,100	13,300	12,000
Antimony	2,260	4,050	3,810
Selenium	< 2,000	< 2,000	< 2,000
Thallium	< 6,000	< 6,000	< 6,000

TABLE C-25. CONCENTRATION OF METALS IN
PARTICULATE EMISSIONS
($\mu\text{g/g}$)^a

Metal	Run 1	Run 2	Run 3
Silver	14.8	11.7	7.6
Arsenic	< 300	< 240	< 300
Barium	1,250	590	690
Beryllium	< 0.40	< 0.32	< 0.41
Cadmium	1,130	630	900
Chromium	2,620	1,610	2,770
Mercury	56	< 44	< 56
Nickel	590	200	420
Lead	91,600	68,900	96,100
Antimony	820	9,130	15,200
Selenium	< 600	< 500	< 600
Thallium	150	120	150

^a Metal concentrations on a blank filter were quite low relative to amounts in actual samples. (All values were below ICP detection limit except Ba, which was in the low ppm range.)

TABLE C-26. MASS RATES FOR METALS

Metal	Input			Effluent			Effluent from		
	from waste feed, g/min			from first absorber, g/min			stack, g/min (% of input)		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Silver	< 0.02	< 0.02	< 0.02	< 3.78	< 3.78	< 3.78	0.001 (> 5)	0.001 (> 5)	0.0006 (> 3)
Barium	59.1	41.9	40.9	48.8	36.3	27.8	0.096 (0.16)	0.054 (0.13)	0.052 (0.13)
Cadmium	9.03	5.52	0.56	24.2	24.0	22.1	0.087 (0.96)	0.058 (1.1)	0.069 (12.3)
Chromium	22.7	20.5	17.0	15.2	14.0	13.9	0.20 (0.88)	0.15 (0.73)	0.21 (1.2)
Nickel	10.7	36.1	12.2	5.54	4.50	8.27	0.045 (0.42)	0.018 (0.05)	0.032 (0.26)
Lead	84.7	66.3	64.2	266	251	227	7.1 (8.4)	6.4 (9.7)	7.3 (11.4)
Antimony	< 0.77	15.8	13.2	42.7	76.5	72.0	0.063 (> 8.2)	0.84 (5.3)	1.2 (9.1)
Thallium	< 0.67	< 0.65	< 0.64	< 113.4	< 113.4	< 113.4	0.012 (> 1.8)	0.011 (> 1.7)	0.011 (> 1.7)
Mercury	< 0.34	< 0.33	< 0.33	< 56.7	< 56.7	< 56.7	0.0043 (> 1.3)	< 0.0041	< 0.0043

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. Performance audits (quantitative evaluations) and systems audits (qualitative evaluations) were conducted for the analysis phases of the project.

Sampling Audit--

A stringent sampling audit consisting of performance and systems audits was conducted on site at the Ross Incinerator facility. Performance audits were conducted on:

- Meter Boxes, Modified Method 5;
- Barometer, aneroid;
- Beckman NDIR;
- Orsat;
- RAC Console Temperature Gauge; and
- Dry Gas Meter.

Systems audits were conducted on:

- Modified Method 5 Train Operation;
- Volatile Organic Sampling Train Operation;
- EPA Standard Methods 1, 2, 3, and 4;
- Process Data Collection;
- Scrubber Effluent Sampling;
- Waste Feed Grab Sampling;
- Orsat Operation; and
- Integrated Gas Bag Sampling.

The performance audits results indicated that there were no problems which affected the quality of the sampling. The audit did detect a malfunctioning meter on Meter Box No. 9. The meter has been replaced. The systems audit indicated no significant problems.

Laboratory Audit--

A comprehensive performance audit and limited systems audit were conducted for the laboratory analysis phase of the project. Performance audit samples were made up and run prior to a particular analysis. Performance audits were conducted on:

- GC/MS analysis of volatile and/or semi-volatile organics;
- ICP analysis of metals; and
- Technicon Autoanalyzer analysis of chlorides.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics and semi-volatile organics; however, the results for the quantitation of bis-ethylhexylphthalate were erratic. This portion of the analytical program is judged to be within control limits.

The analysis of metals performance audit samples by ICP were adequate and acceptable indicating that this aspect of the analytical phase is in control.

Analysis of chloride audit samples were determined to be adequate and acceptable. This part of the analytical phase is also in control.

Sampling QC Activities

In general, sampling QC activities are scrutinized in that all equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities. Moreover, field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function is proceeding in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures excluded spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure was to monitor the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX D

SUMMARY OF RESULTS FROM TESTING AT
PLANT B

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration. Emissions data for the RIA are being provided by a sampling and analysis program. EPA selected for emission measurements the hazardous waste incinerator at Plant B.

Five test runs were conducted at this site during the period July 21 to 26, 1982. Samples of waste feeds and gaseous, liquid, and solid effluents were collected and analyzed. The following sections of this appendix present a summary of results, a brief process description, a synopsis of the test plan and sampling and analysis methods, a discussion of the test results, and a quality assurance summary.

SECTION 2

SUMMARY OF RESULTS

The major objectives on this test program were to identify and quantify principal organic hazardous constituents (POHCs) in the waste feeds, and the POHCs and products of incomplete combustion (PICs) in the inlet gas to the emission control system and the outlet gas from that system. POHCs were also quantified in ash and scrubber effluents. Particulate emissions, HCl removal efficiency, and metal emissions were also determined.

The compounds identified as POHCs and PICs were divided into two groups: volatiles and semivolatiles. POHCs in the waste feeds and stack gases were:

Volatiles

- Chloroform
- Carbon tetrachloride
- Trichloroethylene
- Tetrachloroethylene
- Toluene

Semivolatiles

- Phenol
- Napthalene
- Diethyl phthalate
- Butyl benzyl phthalate

PICs in the effluent gas were:

Volatiles

- Benzene

Semivolatiles

- m-Dichlorobenzene
- p-Dichlorobenzene
- o-Dichlorobenzene
- 1,2,4-Trichlorobenzene
- Dimethyl phthalate
- Hexachlorobenzene

The two volatile POHCs found at the highest concentrations in the gas effluent samples were chloroform and carbon tetrachloride. Because the quantification of these and other volatile POHCs was complicated by significant blank values for several gas bags and volatile organic sampling

train (VOST) traps, the volatile POHC results should be viewed cautiously. In this appendix, data on emission of POHCs are presented as uncorrected concentrations and emission rates accompanied by results for all "blanks."

Results of the particulate measurements on this test program showed that the average particulate concentration (corrected to 7% O₂) for normal operation during Runs 2 and 3 was 400 mg/dscm (0.174 gr/dscf), which exceeds the value in EPA regulations for hazardous waste incinerators of 180 mg/dscm (0.08 gr/dscf). Chloride removal efficiency averaged 98.4% for Runs 1 through 3, which is less than the required removal efficiency of 99.0%. Measurable concentrations of several metals were found in the waste feed. Those metals which showed the highest emission rates in the stack particulate were lead, selenium, and chromium.

All of these results are presented in detail in Section 4, but this appendix contains minimal interpretation of the results.

SECTION 3

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

A description of the sampling procedure and the subsequent analysis performed on the samples is provided in Table D-1 for each sampling point. All collected samples that were not analyzed and the portions of samples remaining after analysis were archived.

Test Runs 1 through 3 included simultaneous collection of samples from the gas stream at the inlet and outlet of the air pollution control system during normal incinerator operation. For test Runs 4 and 5, where conditions were purposely altered from normal operation, the control system outlet gas was not sampled.

Aqueous and organic wastes fed continuously to the incinerator were sampled every 15 min during the test runs to generate a composite sample of each waste. A 50/50 mixture (by volume) of carbon tetrachloride and trichloroethylene was continuously pumped (spiked) into the organic waste feed line, at a rate of about 75 mL/min.

The method of sampling effluents consisted of grab sampling, except for the stack. Stack sampling was accomplished using a Modified Method 5 (MM5) train for particulates, chloride, metals, and semivolatile POHCs; two methods for sampling volatile POHCs (gas bags and VOST traps); and continuous monitors for O₂, CO₂, CO, and total HC. On one test day, fugitive hydrocarbon sampling was also done; these data were retained by EPA personnel who took part in the fugitive sampling.

A schematic diagram of the MM5 train is shown in Figure D-1, and a diagram of the VOST system is shown in Figure D-2.

Gas bag samples were collected in 15-L Tedlar bags by drawing gas into the bags at a rate of about 150 mL/min over a period of about 1 hr. Gas bags filled in the field with prepure N₂ were prepared as gas bag blanks. In the field after the gas bag samples were collected, 5 L of gas was drawn through a Tenax/charcoal trap. At the analytical laboratory, these traps were analyzed along with similar samples prepared at the lab by drawing the contents of the gas bags into Tenax/charcoal traps. The purpose of analyzing gas bag samples transferred onto Tenax/charcoal traps both in the field and at the lab was to protect against the possible loss of gas bag samples due to leakage of the sample out of the bag, and to assess possible contamination of bag samples during shipment to the lab.

TABLE D-1. SUMMARY OF SAMPLING AND ANALYSIS

Sampling point	Number of samples per run	Sampling method	Analysis
<u>Waste feeds</u>			
1. Organic waste feed line	1 composite	Grab sample every 15 min	Heating value POHCs, ultimate Cl, ash H ₂ O 12 metals
2. Aqueous waste feed line	1 composite	Grab sample every 15 min	As above and viscosity
<u>Ash^a</u>			
3. Ash from particulate control device	1 composite	1 to 3 grab samples as available	POHCs Cl ⁻ 12 metals EP toxicity
<u>Scrubber liquids^b</u>			
4. Makeup water to scrubber	1 grab	1 grab sample	Chloroform Cl ⁻ POHCs
5. Scrubber effluent	1 composite	3 grab samples	pH POHCs Cl ⁻
<u>Inlet gas^c</u>			
6. Inlet duct	1	MM5 train	Particulate weight 12 metals on par- ticulate Semivolatile POHCs and PICs Cl ⁻
	1	VOST	Volatile POHCs and PICs
	1	Gas bag	Volatile POHCs and PICs
	1	Orsat	CO ₂ and O ₂

(continued)

TABLE D-1. (continued)

Sampling point	Number of samples per run	Sampling method	Analysis
<u>Stack^d</u>			
7. Outlet stack	1	MM5 train	Particulate weight 12 metals in particulate Semivolatile POHCs and PICs Cl
	1	VOST	Volatile POHCs and PICs
	1	Gas bag	Volatile POHCs and PICs
	1	Orsat	CO ₂ and O ₂
	Continuous	Continuous monitors	CO ₂ , CO, O ₂ , HC
<u>Exhaust air from particulate control device^e</u>			
8. Stack	1	M5 train	Particulate weight
	1	Gas bag	Volatile POHCs

- a EP toxicity was performed for Run 2.
- b Scrubber effluent samples were collected at a point preceding the caustic treatment of the effluent from the plant. Makeup water was analyzed for chloroform in Run 2 and POHCs in Run 5.
- c Metals were analyzed only in Run 2. VOST samples were analyzed only in Runs 2 and 4.
- d Outlet stack sampling was conducted only during Runs 1, 2, and 3. However, the continuous gas monitoring and Orsat testing were performed during all runs. VOST samples were analyzed only in Run 2.
- e The exhaust air was sampled by Method 5 only during Runs 2 and 3. The gas bag sample was taken on Run 3 only.

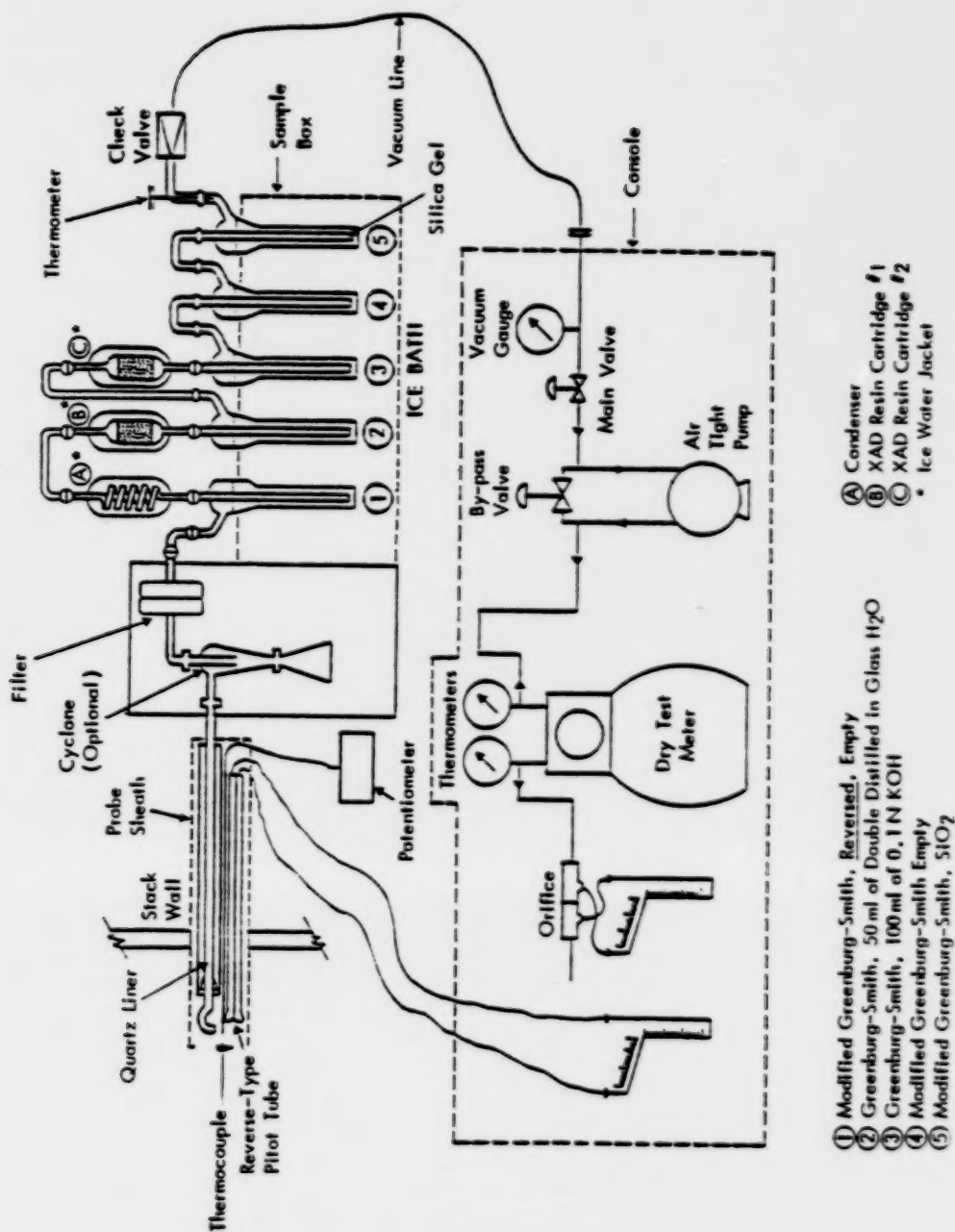


Figure D-1. Modified Method 5 sampling train (MN5).

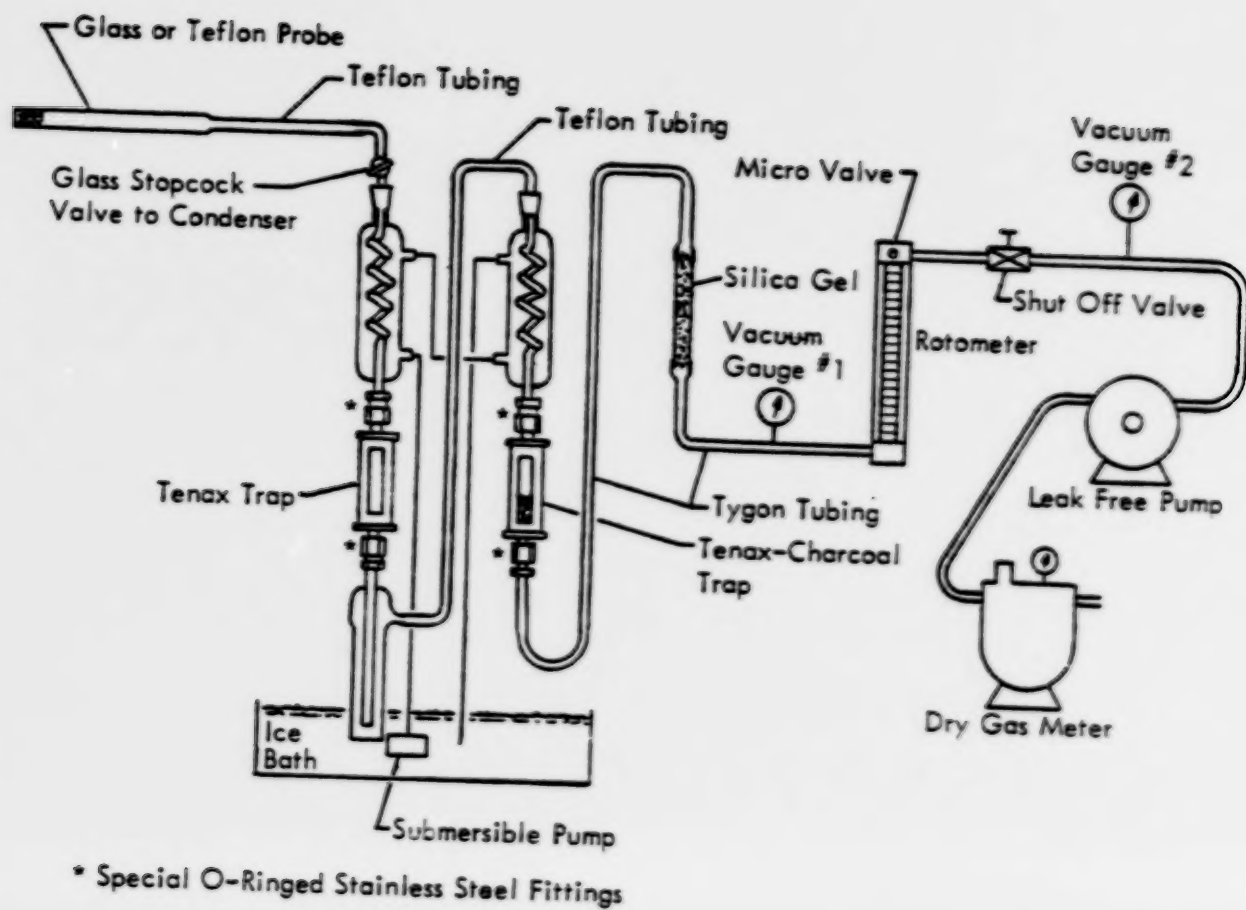


Figure D-2. Volatile organic sampling train (VOST).

In addition to gas bags, the VOST sampling system was also used to sample volatile POHCs. The VOST system consists of a Tenax trap and a backup Tenax/charcoal trap. Gas was drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The first pair of traps was then removed and replaced with another pair, until a total of six pairs were collected (i.e., 120 min of sampling time). Field blank samples which were exposed to the sampling location for the same period required to insert sample traps into the VOST apparatus were also collected. All traps were then returned to the analytical laboratory for analysis of volatile POHCs.

POHCs in the waste feed and stack samples were determined by gas chromatography/mass spectrometry (GC/MS). Prior to analysis, the waste feed samples were homogenized either manually or mechanically. The homogenized feed samples were analyzed for volatile POHCs using a protocol based on Method A101b in the A. D. Little report, "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0-g aliquot of the mixed waste feed sample was dispersed in 40 mL of prepurified tetraglyme. A 10- μ l aliquot of the tetraglyme solution was spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample was then analyzed by the purge-trap-desorb technique described in the A. D. Little report Method A101A (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis may be repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs were analyzed in the waste feed samples according to Method P023 in the A. D. Little report. A 5-g portion of the homogenized waste feed sample was spiked with semivolatile surrogate compounds such as decafluorobiphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 μ g/g, depending on the expected sample complexity. The sample was allowed to equilibrate with the spiked compounds and then was extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appeared on the surface of the methylene chloride, the pH was checked and the sample neutralized, if necessary. The extract was then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

An aliquot of the extract was then screened by gas chromatography (GC) with a flame ionization detector. If necessary, the extract was further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column. A 1.0-mL aliquot of the extract was then spiked with 20 μ g of D₁₀-anthracene internal standard and analyzed by fused silica capillary column GC/MS.

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with VOST. A known amount of the gas bag sample, usually 5.0 L, was transferred onto a Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). A needle was inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were then analyzed using the same techniques that are described below for the VOST traps.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene internal standards using the flash vaporization technique, in which the spiking solution is vaporized and carried onto the trap with a carrier gas. The glass traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, was turned on through the trap and a syringe needle injected through the GC septum port. Slowly, 1 µL of a 25-ng/µL methanolic solution of the internal standards was expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was only about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL), which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds passed into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs in the stack involved several components of the MM5 train. Each of the components of the MM5 train was spiked with a separate surrogate standard according to the following scheme:

Front XAD-2 resin trap - 20 µg of ¹³C-tetrachlorobiphenyl
Back XAD-2 resin trap - 20 µg of decafluorobiphenyl
Particulate filter plus probe rinse residue - 20 µg of D₁₂-chrysene
Condensate water - 20 µg of D₈-naphthalene

The spiked components were normally extracted separately and the extracts combined, dried with anhydrous sodium sulfate, concentrated to 1.0 mL using Kuderna-Danish evaporation according to Method P031 in the A. D. Little report and using supplementary gentle nitrogen blowdown, and analyzed by fused silica capillary column GC/MS. The samples were extracted according to the following procedures:

XAD-2 resin traps: The XAD-2 resin traps were Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Particulate filters plus probe rinse residues: The residue from the acetone rinse of the probe was added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Condensate water: The condensate water was extracted with three 60-mL aliquots of methylene chloride as described in Method P021a in the A. D. Little report (equivalent to EPA Method 625).

POHCs in scrubber water and ash samples were analyzed using the procedures described above. To analyze for metals, the samples were acid digested and analyzed by inductively coupled argon plasma emission spectrophotometry and/or atomic absorption spectrophotometry. Particulate and chloride analyses were conducted according to EPA Method 5 and EPA Method 325.2, respectively, from "Methods for Chemical Analysis of Water and Wastes." EP toxicity samples were tested according to Federal Register, 45, May 19, 1980. Water content in the waste feeds was determined by the Karl Fischer titration method. Chlorine in the waste feed and fabric filter ash was determined by potentiometric titration using a Fisher Titralyzer Model 41. Heating value was determined by ASTM Methods D240-64 and D-2382. Orsat analyses were performed according to EPA Method 3. Continuous monitoring involved the following:

- O₂ - Beckman Model 742 (polarographic sensor)
- CO - Beckman Model 215A (nondispersive infrared)
- HC - Beckman Model 402 (heated oven flame ionization detector)
- CO₂ - Horiba Model PIR-2000S (nondispersive infrared)

In Appendix B, descriptions of all the above sampling and analysis methods are presented in more detail.

SECTION 4

DISCUSSION OF RESULTS

INPUT AND OUTPUT CONCENTRATIONS AND RATES FOR POHCs

POHC input and output concentrations and mass rates were determined for all POHCs listed by EPA which were detected in the waste feeds at concentrations above 100 $\mu\text{g/g}$. No attempt was made to restrict the number of POHCs based on any selection or ranking method. All POHCs reported are compounds that were quantified against authentic standards.

Table D-2 shows the waste feed rates during each run, and the concentrations of POHCs in the waste feeds are given in Table D-3. Input rates for the POHCs are presented in Table D-4, based upon the POHC concentration values in Table D-3 and the waste feed rates presented in Table D-2.

Tables D-5 and D-6 present volatile POHC output data (not blank corrected) for both gas bags and VOST samples. Table D-5 lists the output concentrations in ng/L , and Table D-6 shows the output rates in g/min . Both tables identify samples from the inlet and outlet of the air pollution control system, as well as gas bag samples transferred onto Tenax traps in the field and at the lab after the bags had been returned to the laboratory.

As noted earlier, gas bag samples were transferred onto Tenax traps both in the field and after the bags were returned to the lab. The purpose of following this protocol was to provide a backup sample in case the bag leaked during shipment and to provide information on potential bag contamination occurring during shipment of the bags to the lab. In several cases it was found that the traps transferred from bags in the field contained higher quantities of POHCs than the traps transferred from bags at the lab. Thus, the desired information on bag contamination during shipment was not obtained.

Blank values for many of the VOST traps and bags were sufficiently high for this test to significantly complicate calculation of volatile POHC emission rates. Thus, the volatile POHC emission results should be viewed cautiously.

Semivolatile POHC data are provided in Tables D-7 and D-8. Table D-7 lists the output concentrations, and Table D-8 gives the output rates. The data are not blank corrected.

TABLE D-2. WASTE FEED RATES, kg/min (lb/min)^a

	Run 1 7/23/82	Run 2 7/24/82	Run 3 7/24/82	Run 4 7/26/82	Run 5 7/26/82
Aqueous waste	19.3 (42.5)	27.9 (61.6)	27.2 (60)	34.0 (75.0)	27.2 (60.0)
Organic waste	15.1 (33.2)	15.3 (33.7)	12.9 (28.5)	12.7 (28)	14.1 (31.1)

^a Waste feed rates calculated from change in feed tank levels during each test run.

TABLE D-3. CONCENTRATION OF POHCs IN WASTE FEED ($\mu\text{g/g}$)

	Organic waste feed					Aqueous waste feed				
	Run 1	Run 2	Run 3	Run 4	Run 5	Run 1	Run 2	Run 3	Run 4	Run 5
Volatile POHCs										
Chloroform	330	170	270	110	180	15	21	22	19	17
Carbon tetra- chloride	3,700 ^b	3,700 ^b	4,400 ^b	4,400 ^b	3,500 ^b	< 10	< 10	< 10	< 10	< 10
Trichloro- ethylene	3,700 ^b	3,700 ^b	4,400 ^b	4,400 ^b	3,500 ^b	60	90	85	78	57
Tetrachloro- ethylene	12,000	7,100	9,800	4,400	7,200	1,030	1,600	1,300	1,500	600
Toluene	54,000	32,000	45,300	20,500	33,000	1,600	2,900	2,700	2,700	2,900
Semivolatile POHCs										
Phenol	1,600	1,500	1,700 ^a	1,200	1,400	1,400	1,800	2,900	2,400	2,000
Naphthalene	340	250	450 ^a	270	300	45	44	49	39	46
Diethyl phthalate	930	620	1,300 ^a	930	970	230	240	240	230	230
Butyl benzyl phthalate	490	120	450 ^a	440	370	23	< 5	8	21	< 5

^a Average of triplicate samples.

^b These values calculated from spiking rates.

TABLE D-4. CALCULATED INPUT RATE FOR POHCs IN WASTE FEEDS (g/min)

	Run 1 7/23/82	Run 2 7/24/82	Run 3 7/24/82	Run 4 7/26/82	Run 5 7/26/82
<u>Volatile POHCs</u>					
Chloroform	5.3	3.2	4.1	2.0	3.0
Carbon tetrachloride	56 ^b	57 ^b	57 ^b	56 ^b	49 ^b
Trichloroethylene	57 ^b	59 ^b	59 ^b	58 ^b	51 ^b
Tetrachloroethylene	200	150	160	110	120
Toluene	850	570	650	350	540
<u>Semivolatile POHCs^a</u>					
Phenol	51	73	100		
Naphthalene	6.0	5.1	7.1		
Diethyl phthalate	18	16	23		
Butyl benzyl phthalate	7.8	1.8	6.0		

^a Semivolatile stack samples were collected only during Runs 1, 2, and 3.

^b These values calculated from spiking rates.

TABLE D-5. CONCENTRATIONS OF VOLATILE PHASES IN STACK GASES (NOT BLANK CORRECTED), ng/L

Gas bags	Run 1, 7/23/82		Run 2, 7/24/82		Run 3, 7/24/82		Run 4, 7/26/82		Run 5, 7/26/82		Run 3, 7/26/82	
	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (field)	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (lab)
Chloroform	12	70	12	64	8	10	18	30	320	230	< 1	1
Carbon tetrachloride	62	40	1F ^c	1F	26	20	1F	140	920	1F	1F	10
Trichloroethylene	40	80	26	30	12	170	340	160	> 620	> 620	1,600	330
Tetrachloroethylene	26	30	32	6	10	8	200	90	380	150	40	7
Toluene	220	40	100	240	190	20	> 500	110	500	80	160	20

VOST	Run 2 ^d			Run 4 ^{a,d}		
	Inlet	Outlet	3rd Pair	Inlet	Outlet	3rd Pair
Chloroform	13	12	10	1F	27	49
Carbon tetrachloride	1F	1F	1F	1F	1F	1F
Trichloroethylene	22	20	21	61	25	260
Tetrachloroethylene	6.7	9.2	11	28	17	460
Toluene	110	100	61	210	430	> 530

- ^a Incineration conditions were purposely altered from normal operation for Run 4 and 5 to obtain data on poor performance.
- ^b Designations in parentheses refer to gas bag samples transferred onto Tenax traps in field and after bags were returned to the lab.
- ^c 1F indicates interference of other compounds which prevented quantitation of compound of interest.
- ^d VOST sampling was conducted only during Run 2 (inlet and outlet) and Run 4 (inlet only).

TABLE D-6. OUTPUT RATES FOR VOLATILE PHTHS (NOT BLANK CORRECTED), g/min

	Run 1, 7/23/82		Run 2, 7/24/82		Run 3, 7/24/82		Run 4, 7/26/82		Run 5, 7/26/82		Run 3, 7/26/82	
	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (field)	Inlet (field)	Inlet (lab)	Inlet (field)	Inlet (lab)	Inlet (field)	Control device air exhaust (field) (lab)
Chloroform	0.0025	0.016	0.0023	0.016	0.0015	0.0019	0.0037	0.0062	0.064	0.046	< 0.00003	0.00003
Carbon tetrachloride	0.0087	0.0092	IF ^c	0.0045	IF	0.0038	IF	0.029	0.18	IF	IF	0.0003
Trichloroethylene	0.0083	0.018	0.0055	0.014	0.0027	0.021	0.071	0.032	> 0.12	> 0.12	0.046	0.010
Tetrachloroethylene	0.0054	0.0069	0.0074	0.0016	0.0014	0.0015	0.043	0.019	0.076	0.029	0.001	0.0002
Toluene	0.046	0.0092	0.024	0.0045	0.054	0.0038	> 0.10	0.023	0.10	0.016	0.005	0.0006

	Run 2 ^d			Run 4 ^d		
	Inlet 3rd Pair	Inlet 6th Pair	Outlet 4th Pair	Inlet 1st Pair	Inlet 3rd Pair	Inlet 6th Pair
Chloroform	0.0026	0.0023	IF	0.0061	0.010	0.0050
Carbon tetrachloride	IF	IF	IF	IF	IF	IF
Trichloroethylene	0.0043	0.0039	0.0041	0.014	0.053	0.011
Tetrachloroethylene	0.0013	0.0018	0.0022	0.0064	0.046	0.026
Toluene	0.022	0.020	0.012	0.049	> 0.071	0.032

- ^a Incubation conditions were purposely altered from normal operation for Runs 4 and 5 to obtain data on poor performance.
- ^b Designations in parentheses refer to gas bag samples transferred onto Texas traps in field and after bags were returned to the lab.
- ^c IF indicates interference of other compounds which prevented quantitation of compound of interest.
- ^d VOST sampling was conducted only during Run 2 (inlet and outlet) and Run 4 (inlet only).

TABLE D-7. CONCENTRATIONS OF SEMIVOLATILE POHCs IN STACK GASES
(NOT BLANK CORRECTED), ng/L

	<u>Run 1, 7/23/82</u>		<u>Run 2, 7/24/82</u>		<u>Run 3, 7/24/82</u>	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Phenol	13	47	13	35	38	11
Naphthalene	7	39	5	42	16	24
Diethyl phthalate	8	36	53	45	43	31
Butyl benzyl phthalate	< 1	3	1	7	3	3

TABLE D-8. OUTPUT RATE FOR SEMIVOLATILE POHCs (NOT BLANK CORRECTED), g/min

	Run 1, 7/23/82		Run 2, 7/24/82		Run 3, 7/24/82	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
Phenol	0.0026	0.011	0.0024	0.0080	0.0076	0.0024
Naphthalene	0.0015	0.0090	0.00097	0.0094	0.0031	0.0052
Diethyl phthalate	0.0016	0.0082	0.010	0.010	0.0080	0.0067
Butyl benzyl phthalate	0.00016	0.00074	0.00022	0.0016	0.00049	0.00061

EMISSION OF PICs

Analysis of samples included identification of PICs, i.e., those compounds identified in the stack samples that are listed as POHCs but which were not found in the waste feed samples at concentrations above 100 µg/g. The output concentration and rate for these compounds are tabulated in Tables D-9 and D-10.

BLANK VALUES FOR POHCs AND PICs

The following tables present the gas bag and VOST blank data for volatile and semivolatile POHCs. Table D-11 gives the average blank values and the standard deviation of the volatile blank population. The sample volumes for gas bags and VOST samples are presented in Table D-12. Blank correction values for semivolatiles are presented in Table D-13. Sample volumes for the MM5 train can be found in Table D-17.

DREs FOR VOLATILES AND SEMIVOLATILES

Destruction and removal efficiencies (DREs) based on the above data are presented in Table D-14. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$\text{DRE} = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

POHCs, CHLORIDE, AND pH IN SCRUBBER WATER STREAMS

The tests included sampling and analysis of the scrubber effluent to determine the concentrations of POHCs and chloride and measurement of the pH. The results are given in Table D-15.

A sample of the city water makeup to the scrubber was also analyzed 4 months after the test and found to contain 35 µg/L of chloroform. At the estimated makeup rate for the city water of 600 L/min, this would amount to 0.021 g/min of chloroform. That amount is greater than the quantities contained in the outlet stack gas, as given previously in Table D-6. This result and the fact that little or no chloroform was detected in the scrubber water discharge samples may explain why chloroform levels were generally higher in the outlet stack gas than in the inlet gas. The data suggest that chloroform is stripped from the makeup water in the scrubber.

The results of the scrubber water analyses can also be used to indicate the degree of POHC removal by the scrubber. In Runs 1 through 4, toluene was the only compound found at a concentration above 1 µg/L. At the highest concentration found of 2.6 µg/L in Run 2, the amount discharged in the scrubber effluent, based on the flow rate of 600 L/min, would be 0.002 g/min.

TABLE D-9. CONCENTRATIONS OF SEMIVOLATILE AND VOLATILE PICs IN STACK GASES (NOT BLANK CORRECTED), ng/L

Semi-volatile PICs	Run 1, 7/23/82		Run 2, 7/26/82		Run 3, 7/26/82	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
m-Dichlorobenzene	2.2	2.8	0.6	5.8	4.9	2.7
p-Dichlorobenzene	1.2	1.5	0.3	3.6	3.2	2.1
o-Dichlorobenzene	2.1	3.3	0.6	6.3	5.3	3.1
1,2,4-Trichlorobenzene	4.5	6.0	1.5	9.0	4.1	5.1
Dimethylphthalate	< 0.4	< 0.7	< 0.4	< 0.5	0.5	1.1
Hexachlorobenzene	8.7	7.9	8.1	10.0	11.0	1.6

Volatile PICs by gas bags	Run 1, 7/23/82		Run 2, 7/26/82		Run 3, 7/26/82		Run 4, 7/26/82		Run 5, 7/26/82	
	Inlet (field) ^a	Outlet (lab)	Inlet (field) ^a	Outlet (lab)	Inlet (field) ^a	Outlet (field) ^a	Inlet (field) ^a	Outlet (lab) ^a	Inlet (field)	Outlet (lab)
Benzene	14	30	48	140	10	18	40	25	> 140	> 140

Volatile PICs by VOST	Run 2				Run 4	
	Inlet	Outlet	1st Pair	4th Pair	1st Pair	3rd Pair
Benzene	16	20	16	> 11	39	16

^a Designation in parentheses refer to locations where gas bag samples were transferred onto Tenax traps.

TABLE D-10. OUTPUT RATES FOR SEMIVOLATILE AND VOLATILE PICs (NOT BLANK CORRECTED), g/min

Semivolatile PICs	Run 1, 7/23/82		Run 2, 7/24/82		Run 3, 7/24/82	
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet
m-Dichlorobenzene	0.00046	0.00065	0.00015	0.0013	0.00094	0.00058
p-Dichlorobenzene	0.00024	0.00035	0.000079	0.0010	0.00061	0.00046
o-Dichlorobenzene	0.00044	0.00075	0.00015	0.0018	0.0010	0.00067
1,2,4-Trichlorobenzene	0.00094	0.0014	0.0028	0.0020	0.00079	0.0011
Bis(2-ethylphthalate)	< 0.000085	< 0.00015	< 0.000084	< 0.00012	0.000088	0.00024
Hexachlorobenzene	0.0018	0.0018	0.0016	0.0023	0.0021	0.00035

Volatile PICs by Run logs	Run 1, 7/23/82		Run 2, 7/24/82		Run 3, 7/24/82		Run 4, 7/26/82	
	Inlet (field) ^a	Outlet (lab) ^a	Inlet (field) ^a	Outlet (field) ^a	Inlet (field) ^a	Outlet (field) ^a	Inlet (field) ^a	Outlet (lab) ^a
Benzene	0.0029	0.0069	0.011	0.027	0.0023	0.0041	0.0034	0.0076
							0.0031	0.012
								0.0052
								> 0.027
								> 0.027

Volatile PICs by VORT	Run 2				Run 4	
	1st Pair	3rd Pair	6th Pair	Outlet 4th Pair	1st Pair	3rd Pair
Benzene	0.0036	0.0045	0.0036	> 0.0021	0.0013	0.0081
						0.0033

^a Designations in parentheses refer to locations where gas bag samples were transferred onto Tenax traps.

TABLE D-11. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>				
Chloroform	< 1	0	77	120
Carbon tetrachloride	10	7.6	245	318
Trichloroethylene	160	53	670	294
Tetrachloroethylene	13	5.8	334	187
Toluene	57	5.8	870	240
<u>PIC</u>				
Benzene	35	13	290	210

TABLE D-12. VOLATILE SAMPLE VOLUMES (DRY STANDARD LITERS)

Gas bags		VOST	
Run No.	Volume (L)	Run No./Pair No.	Volume (L)
All gas bag samples were analyzed using a sample volume of 5.0 L		Inlet 2/1	19.4
		2/3	19.5
		2/6	19.9
		Outlet 2/1	19.6
		2/4	18.4
		Inlet 4/1	17.8
		4/3	16.2
		4/6	20.0

TABLE D-13. SEMIVOLATILE BLANK CORRECTION
VALUES

Compound	Blank correction value (µg)
<u>POHCs</u>	
Phenol	0.7
Naphthalene	< 1
Diethyl phthalate	7.7
Butyl benzyl phthalate	1.7
<u>PICs</u>	
m-Dichlorobenzene	< 1
p-Dichlorobenzene	< 1
o-Dichlorobenzene	< 1
1,2,4-Trichlorobenzene	< 1
Dimethyl phthalate	0.1
Hexachlorobenzene	< 1

TABLE D-14. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs)^a (BLANK CORRECTED)

Volatile compounds	Gas bags									
	Run 1		Run 2		Run 3		Run 4 ^b		Run 5 ^b	
	Inlet (field) ^c	Outlet (lab)	Inlet (field)	Outlet (lab)	Inlet (field)	Outlet (field)	Inlet (field)	Outlet (field)	Inlet (field)	Outlet (lab)
Chloroform	99.953	99.70	99.927	99.50	99.963	99.953	99.81	99.69	97.9	98.5
Carbon tetrachloride	99.985	99.984	IF	99.9928	99.9919	99.9940	IF	99.949	99.63	IF
Trichloroethylene	> 99.985	99.981	> 99.990	> 99.977	99.975	99.955	< 99.80	99.956	< 99.80	> 99.80
Tetrachloroethylene	99.9976	99.9968	99.9966	99.9993	99.99918	99.9986	99.99918	99.9962	99.983	99.937
Toluene	99.9749	99.9923	99.9975	99.9949	99.9944	99.9975	< 99.97	99.9962	99.982	99.997
PM5										
Semivolatile compounds	Run 1	Run 2		Run 3						
Phenol	99.979	99.989								
Naphthalene	99.85	99.81								
Diethyl phthalate	99.962	99.943								
Butyl benzyl phthalate	99.978	99.92								

IF = Quantification of this compound prohibited by interfering peak on GC/MS.

^a Blank values for many of the VOST traps and bags were sufficiently high to complicate quantification of emission rates; thus the volatile PM10 DREs should be viewed cautiously.

^b Incineration conditions were purposely altered from normal operation for Runs 4 and 5 to obtain data on poor performance. The combustion temperatures for Runs 4 and 5 were 960°C (1776°F) and 956°C (1753°F), respectively, versus an average temperature over Runs 1, 2, and 3 of 1067°C (1952°F).

^c Designations in parentheses refer to gas bag samples transferred onto Tenax traps in field and after bags were returned to the lab.

TABLE D-15. POHC AND CHLORIDE CONCENTRATIONS IN SCRUBBER WATER SAMPLES

	Run 1	Run 2	Run 3	Run 4	Run 5
<u>Volatile POHCs (µg/L)</u>					
Chloroform	< 1	< 1	< 1	< 1	1
Carbon tetrachloride	< 1	< 1	< 1	< 1	< 1
Trichloroethylene	< 1	< 1	< 1	< 1	< 1
Tetrachloroethylene	< 1	< 1	< 1	< 1	< 1
Toluene	1.6	2.6	1.3	1.4	< 1
<u>Semivolatile POHCs^a (µg/L)</u>					
Phenol				0.1	0.7
Naphthalene				< 1	35
Diethyl phthalate				< 2	< 2
Butyl benzyl phthalate				< 1	< 1
<u>Chloride (mg/mL)</u>	1.85	2.34	1.97	1.34	1.95
<u>pH</u>	2	2	2		

^a Only scrubber water samples from Runs 4 and 5 were analyzed for semi-volatile POHCs.

Comparing this with the toluene input rate in the waste feed of 570 g/min (from Table D-4), it is estimated that removal of toluene in the scrubber would be no more than 0.0004% of the input. This is very small relative to the amount combusted in the incinerator.

Naphthalene was detected at a concentration of 35 µg/L in the scrubber water sample from Run 5, equivalent to 0.03 g/min. Assuming that the waste feed input rate for naphthalene during Run 5 was about the same as in Runs 1 through 3, the input rate would be about 6 g/min. In this case, the amount removed by the scrubber is about 0.5% of the input. However, operating conditions were purposely changed during Run 5 to create poor combustion conditions.

POHCs IN ASH

Control device ash samples from each test run were analyzed, and results are given in Table D-16.

PARTICULATE EMISSIONS

The particulate emission data are presented in Table D-17. Associated Method 5 test data are given in Table D-18. The average outlet particulate concentration for Runs 2 and 3 was 400 mg/dscm (corrected to 7% O₂) compared to 180 mg/dscm in EPA's regulation. The average particulate removal efficiency of the control system was 85%.

The total particulate emissions from the stack and the control device air exhaust, as measured in Run 3, were 3.92 kg/hr.

CONTINUOUS MONITORING DATA

Continuous monitors were used to determine concentrations of O₂, CO₂, CO, and HC in the stack effluent throughout the test period. The results are summarized in Table D-19. The concentrations of HC and CO were low except for the high values in Run 5, during which operating conditions were purposely altered to create poor combustion conditions.

HCl EMISSIONS AND REMOVAL EFFICIENCY

Table D-20 summarizes chloride emissions in the inlet and outlet gases. The data show that for Runs 1 and 2 the output rate of chloride was less than the 1.8 kg/hr requirement in current EPA regulations. In Run 3, the chloride output rate was greater than 1.8 kg/hr and the removal efficiency was below the 99% removal efficiency specified in the regulations.

ANALYSES OF WASTE FEEDS AND ASH

Table D-21 summarizes data on percent chlorine, percent water, and heating value for the organic and aqueous waste feeds. The percent chlorine in the control device ash is also listed. Viscosity was determined for the aqueous waste and an ultimate analysis was performed for both waste streams.

TABLE D-16. POHC CONCENTRATIONS IN CONTROL DEVICE
ASH SAMPLES ($\mu\text{g/g}$)

	Run 1	Run 2	Run 3	Run 4	Run 5
<u>Volatile POHCs</u>					
Chloroform	< 1	< 1	< 1	< 1	< 1
Carbon tetrachloride	< 1	1	< 1	< 1	< 1
Trichloroethylene	1	2	< 1	< 1	< 1
Tetrachloroethylene	2	< 1	< 1	2	< 1
Toluene	14	4	4	13	6
<u>Semivolatile POHCs</u>					
Phenol	< 1	< 1	< 1	< 1	< 1
Naphthalene	< 1	< 1	< 1	< 1	< 1
Diethyl phthalate	< 1	< 1	< 1	< 1	< 1
Butyl benzyl phthalate	< 1	< 1	< 1	< 1	< 1

TABLE D-17. PARTICULATE EMISSION DATA^a

	Particulate concentration, mg/dscm (gr/dscf)		Particulate emissions, kg/hr (lb/hr)	
	Run 2	Run 3	Run 2	Run 3
<u>Inlet</u>				
Measured	2,516 (1.097)	2,263 (0.987)	29.38 (64.72)	25.88 (57.01)
Corrected ^b	3,202 (1.396)	2,576 (1.123)	-	-
<u>Outlet</u>				
Measured	340 (0.148)	294 (0.128)	4.63 (10.20)	3.85 (8.47)
Corrected ^b	429 (0.187)	371 (0.161)	-	-
<u>Particulate removal efficiency (%)</u>	-	-	84.2	85.1
<u>Control device air exhaust measured</u>	c	38 (0.017)	c	0.068 (0.15)

^a Values from Run 1 were not reported because loss of sample was suspected when filters got wet during shipment.

^b Measured emissions were corrected to 7% O₂ as follows:

$$C_{\text{corr}} = \frac{(14)}{(21 - \% \text{ O}_2)} (C_{\text{measured}})$$

^c Testing was done only during Run 3 and was single point sampling.

TABLE D-18. METHOD 5 TEST DATA

	Run 1		Run 2		Run 3				
	Inlet	Outlet	Inlet	Outlet	Inlet	Outlet	Air ^a exhaust	Run 4 ^b Outlet	Run 5 ^b Outlet
Volume of gas sampled (Nm ³)	2.422	1.526	2.334	1.898	2.163	2.535	2.239	N/A	N/A
Sampling time (min)	156	160	156	160	156	160	120	N/A	N/A
Percent iso-kinetic	100.1	85	102.6	106.8	97.1	101.9	101.6	N/A	N/A
Moisture content (%)	21.5	14.7	26.8	15.7	27.7	15.1	1.7	N/A	N/A
Percent O ₂ (dry)	10.3	12.7	10.0	9.9	8.7	9.9 ^c	20.9	15.7	12.3
Percent CO ₂ (dry)	10.7	5.1	12.0	7.7	10.2	7.7 ^c	0.0	5.8	8.5
Gas temperature (°C)	236	53	239	55	235	54	116	39	49
Gas flow rate (Act. m ³ /min)	469	308	476	309	468	293	40	277	282
Gas flow rate (Nm ³ /min)	207	230	195	227	191	218	29	236	226

^a Due to small stack size, only single point sampling was conducted.

Outlet data for Runs 4 and 5 are based only on velocity traverse of stack. No HMF sampling was done in these two runs.

^c No Orsat data available. Data from previous run (same day) were used.

TABLE D-19. CONTINUOUS MONITORING DATA (OUTLET)^a

	Run 1 7/23/82	Run 2 7/24/82	Run 3 7/24/82	Run 4 7/26/82	Run 5 7/26/82
Oxygen (%)					
Range	7.1-14.2	9.8-10.8	10.1-18.1	13.3-17.4	9.8-10.9
Average	11.8	10.3	10.7	14.3	10.1
Carbon dioxide (%)					
Range	6.1-8.1	8.0-8.7	4.4-8.6	2.7-5.6	7.7-8.2
Average	6.7	8.2	8.0	4.8	8.0
Carbon monoxide (ppm) ^b					
Range	10.6-22.5	< 1	3.7-9.8	5.2-8.7	2681-4278
Average	12.9	< 1	6.8	6.5	3347
Total hydrocarbons (ppm) ^b					
Range	< 1-1.3	< 1	< 1.0-3.2	< 1.0-3.2	163-535
Average	< 1	< 1	< 1	< 1	277

^a Concentrations on dry gas basis.

^b Total hydrocarbons reported as propane.

TABLE D-20. CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

	Run 1, 7/23/82	Run 2, 7/24/82	Run 3, 7/24/82
Cl in feed			
kg/hr	54.7	69.8	50.8
(lb/hr)	(120)	(154)	(112)
Cl ⁻ in inlet gas to gravel filter			
kg/hr	59.5	73.7	63.2
(lb/hr)	(131)	(163)	(139)
Cl ⁻ out stack			
kg/hr	0.29	0.83	2.03
(lb/hr)	(0.65)	(1.8)	(4.5)
Cl ⁻ removal efficiency, % (based on inlet gas) ^a	99.5	98.9	96.8
Cl removal efficiency, % (based on feed) ^b	99.5	98.8	96.0

^a Calculation based on Cl⁻ measured in inlet and outlet gas samples.

^b Calculation based on Cl⁻ measured in waste feed and outlet gas samples.

TABLE D-21. ANALYSES OF WASTE FEEDS AND ASH

	Run 1 7/23/82	Run 2 7/24/82	Run 3 7/24/82	Duplicate (Run 3)
<u>Chlorine, %</u>				
Organic waste	5.40	6.57	5.37	(6.03)
Aqueous waste	0.50	0.57	0.41	(0.51)
Control device ash	3.98	4.51	4.92	
<u>Water, %</u>				
Organic waste	34.65	37.66	47.65	(43.11)
Aqueous waste	94.36	95.18	96.90	(94.43)
<u>Heating value, kJ/kg (Btu/lb)</u>				
Organic waste	19,750 (8,505)	20,390 (8,780)	19,700 (8,482)	20,090 (8,652)
Aqueous waste	4,090 (1,761)	4,480 (1,930)	4,510 (1,944)	4,420 (1,905)
<u>Ash, %</u>				
Organic waste	4.92	4.82	4.66	
Aqueous waste	1.18	1.07	1.05	(1.08)
<u>Viscosity^a (SUS at 100°F)</u>				
Aqueous waste	-	28.5	33.6	-

Ultimate Analysis	Run	Percent C	Percent H	Percent N	Percent S	Percent O (by difference)
Organic waste	1	42.09	9.31	1.12	0.35	36.81
	2	41.04	9.27	1.05	0.32	36.93
	3	43.60	9.09	1.10	0.33	35.85
Aqueous waste	1	9.05	10.06	0.37	0.033	78.81
	2	8.84	10.00	0.38	0.34	78.80
	3	9.15	10.24	0.36	0.051	78.74

^a Viscosity could not be determined for the organic waste because it gelled.

METALS

Twelve metals were analyzed in waste feed samples from all five runs, and results are given in Table D-22. Control device ash samples from Run 2 were also analyzed for the same metals, using two procedures. In the first procedure, the ash sample was digested in nitric acid. In the second procedure, the ash was leached according to the EP toxicity method, and the leachate analyzed for metals. Both results are given in Table D-23. Data in Table D-23 show that cadmium was the only metal present in the leachate at a concentration greater than the EP toxicity limit. It was not possible to determine if selenium exceeded the limit because of the high detection limit of the analytical method used.

Both the inlet and outlet particulate samples from Run 2 were analyzed for metals; results are given in Table D-24. These results were then used to compute mass emission rates for the metals in the particulate emissions for comparison with metals input in the waste feeds from Run 2, as shown in Table D-25. The data show that lead, selenium, and chromium were emitted in the largest quantities from the outlet (stack).

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (1) external auditing; (2) sampling QA activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. Performance audits (quantitative evaluations) and systems audits (qualitative evaluations) were conducted for the analysis phases of the project.

Sampling Audit--

A stringent sampling audit consisting of performance and systems audits was conducted on site at the incinerator facility. Performance audits were conducted on:

- Meter boxes, Modified Method 5;
- Barometer, aneroid;
- Beckman NDIR;
- Orsat;
- RAC console temperature gauge; and
- Dry gas meter.

TABLE D-22. CONCENTRATION OF METALS IN WASTE FEEDS ($\mu\text{g/g}$)

	Organic waste					Aqueous waste				
	Run 1	Run 2	Run 3	Run 4	Run 5	Run 1	Run 2	Run 3	Run 4	Run 5
Silver	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Arsenic	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20
Barium	110	110	140	90	220	6.3	5.9	6.7	5.3	6.6
Beryllium	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
Cadmium	5.5	5.3	5.6	4.7	7.8	< 1	< 1	< 1	< 1	< 1
Chromium	51	50	57	40	83	2.8	2.6	2.9	2.7	2.7
Mercury	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Nickel	6.7	6.8	7.2	5.6	10.3	2.4	2.2	2.3	2.3	1.9
Lead	140	140	150	120	200	11	< 10	11	< 10	< 10
Antimony	59	58	61	45	87	< 10	< 10	< 10	< 10	< 10
Selenium	120	340	< 100	220	330	< 100	< 100	< 100	< 100	< 100
Thallium	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20

TABLE D-23. CONCENTRATION OF METALS IN CONTROL DEVICE
ASH SAMPLE (RUN 2)

	Concentration in ash sample ^a (µg/g)	Concentration in ash sample leachate ^b (mg/L)	Maximum concen- tration limit in leachate for EP toxicity ^c (mg/L)
Silver	< 32	0.13	5.0
Arsenic	< 100	< 0.03	5.0
Barium	1,600	0.17	100
Beryllium	< 7	< 0.002	d
Cadmium	130	2.0	1.0
Chromium	480	0.2	5.0
Mercury	< 200	0.2	0.2
Nickel	90	0.2	d
Lead	3,100	< 0.04	5.0
Antimony	< 400	0.8	d
Selenium	< 17,000	< 30	1.0
Thallium	< 180	0.1	d

^a Ash samples were digested in nitric acid.

^b Ash samples were leached according to EP toxicity procedures in Federal Register, May 19, 1980.

^c Federal Register, May 19, 1980, p. 33122.

^d These metals are not included in the EP toxicity test.

TABLE D-24. CONCENTRATION OF METALS IN
PARTICULATE ($\mu\text{g/g}$)^a

	Run 2	
	Inlet	Outlet
Silver	< 22	< 200
Arsenic	< 72	< 650
Barium	250	930
Beryllium	5	< 43
Cadmium	140	150
Chromium	950	1,500
Mercury	< 75	< 680
Nickel	650	740
Lead	3,100	5,300
Antimony	300	< 2,500
Selenium	9,200	3,700
Thallium	< 120	< 1,100

^a Metal concentrations in particulate were determined only for samples from Run 2.

TABLE D-25. MASS RATES FOR METALS IN RUN 2
(g/min)^a

	Input in waste feeds			Emissions		Control device ^b ash
	Organic	Aqueous	Total	Inlet	Outlet	
Silver	< 0.02	< 0.03	< 0.05	< 0.011	< 0.015	< 0.01
Arsenic	< 0.30	< 0.60	< 0.90	< 0.035	< 0.050	< 0.03
Barium	1.7	0.16	1.9	0.12	0.072	0.5
Beryllium	< 0.02	< 0.03	< 0.05	< 0.002	< 0.003	< 0.002
Cadmium	0.08	< 0.03	< 0.11	0.069	0.012	0.04
Chromium	0.77	0.07	0.84	0.47	0.120	0.1
Mercury	< 0.20	< 0.30	< 0.50	< 0.037	< 0.052	< 0.06
Nickel	0.10	0.06	0.16	0.32	0.057	0.03
Lead	2.1	< 0.30	2.1	1.5	0.410	0.9
Antimony	0.89	< 0.30	0.89	0.17	< 0.190	< 0.1
Selenium	5.2	< 2.8	5.2	4.5	0.290	< 5.1
Thallium	0.30	< 0.60	< 0.90	< 0.059	< 0.085	< 0.05

^a Metals in particulate emissions were determined only for samples from Run 2.

^b Mass rates for control device ash are based on limited measurements of ash discharge quantities which averaged approximately 300 g/min.

Systems audits were conducted on:

- Modified Method 5 train operation;
- Volatile organic sampling train operation;
- EPA Standard Methods 1, 2, 3, and 4;
- Process data collection;
- Scrubber effluent sampling;
- Waste feed grab sampling;
- Orsat operation; and
- Integrated gas bag sampling.

The results of the performance audits indicated that there were no problems which affected the quality of the sampling. The systems audit indicated no significant operational problems.

The auditor did observe the use of a screwclamp on a length of Tygon tubing to control dilution ratios on the Century organic vapor analyzer used for fugitive emission sampling. Although this procedure was specified in the manual for the instrument, a critical orifice or other equivalent flow control device should be used in place of this screw clamp. Since the sampling supervisor reported that dilution sampling was not used, there was not any impact on the validity of the data collected.

Laboratory Audit--

A comprehensive performance audit and limited systems audit were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on:

- GC/MS analysis of volatile and/or semivolatile organics; and
- Technicon Autoanalyzer analysis of chlorides.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics and semivolatile organics; however, the results for the quantitation of bis(2-ethylhexyl)phthalate were erratic. This portion of the analytical program is judged to be within control limits, however.

Analyses of chloride audit samples were determined to be adequate and acceptable. This part of the analytical phase is also within control limits.

Systems audits demonstrated that laboratory analyses were done according to protocol.

Sampling QA Activities

In general, sampling QA activities are scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures excluded spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure was to monitor the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX E

SUMMARY OF RESULTS FROM TESTING AT
THE UPJOHN INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration which requires the collection of data on current incineration operating practices. Through a comprehensive sampling and analysis program at selected incineration facilities, test data regarding waste feed composition, incinerator effluents, and operating parameters are being acquired.

The EPA selected the hazardous waste incinerator at the Upjohn Company in LaPorte, Texas, as one facility for the test program. On-site sampling was conducted on August 12 and 13, 1982. Samples of the gaseous and liquid waste feeds, the scrubber input and output waters, and the stack effluent were collected and analyzed. The following sections of this appendix present a summary of the emission measurement results, a brief process description, a synopsis of the test plan and the sampling and analysis methods used, and a discussion of the test program results.

SECTION 2

SUMMARY OF RESULTS

The major emphasis of the Upjohn test program was the determination of principal organic hazardous constituents (POHCs) in the waste feeds and the stack effluent. The results of these analyses, including a summary of destruction and removal efficiencies (DREs), are presented in Section 5. The compounds shown in Table E-1 were identified either as POHCs (i.e., greater than 100 $\mu\text{g/g}$ in the waste materials) or were cited as products of incomplete combustion (PICs) in the stack effluent (not found in the waste feeds). Phosgene, which is not a POHC, is included because it was a major component of the waste.

Table E-2 presents a summary of the emissions of chlorides and particulates. The results shown are averages of the data from three sampling runs. The average chloride removal efficiency was 99.75%. Particulate emissions, corrected to 7% O_2 , averaged 200 mg/dscm (0.087 gr/scf), a value greater than the federal particulate emission limitation of 180 mg/dscm (0.08 gr/dscf) for hazardous waste incinerators.

All of the test results are presented in detail in Section 5. This appendix contains minimal interpretation of the results.

TABLE E-1. SUMMARY OF POHCs AND PICs IDENTIFIED AT UPJOHN

POHCs	PICs
<u>Volatile compounds</u>	
Carbon tetrachloride ^a	Chloroform
Trichloroethylene ^a	Benzene
Chlorobenzene	Tetrachloroethylene
Chloromethane	Toluene
	Methylene chloride
	Methyl ethyl ketone
	Bromodichloromethane
	Dibromochloromethane
<u>Semivolatile compounds</u>	
<u>m</u> -Dichlorobenzene	Phenol
<u>o</u> -Dichlorobenzene	Naphthalene
<u>p</u> -Dichlorobenzene	2,6-Toluene diisocyanate
1,2,4-Trichlorobenzene	Diethyl phthalate
Phosgene ^b	Hexachlorobenzene
Aniline	<u>o</u> -Chlorophenol
Phenyl isocyanate	2,4,6-Trichlorophenol
Chlorophenyl isocyanate	Pentachlorophenol
Bis(ethylhexyl)phthalate	<u>o</u> -Nitrophenol

^a These two compounds were introduced into the liquid waste stream using an injection system.

^b Phosgene does not appear on EPA's Appendix VIII listing of organic hazardous constituents. However, it is the major component of the vent gas waste stream and it is a hazardous chemical.

TABLE E-2. AVERAGE STACK EFFLUENT SAMPLING RESULTS

Chlorides^a

Emission rate, g/min (lb/hr)	9.6 (1.3)
Removal efficiency, %	99.75

Particulates

Emission rate, mg/dscm (gr/dscf)	181.1 (0.0791) ^b
Corrected to 7% O ₂	199.7 (0.0872) ^b

^a Total chlorides expressed as HCl.

^b Average of two runs only.

SECTION 3

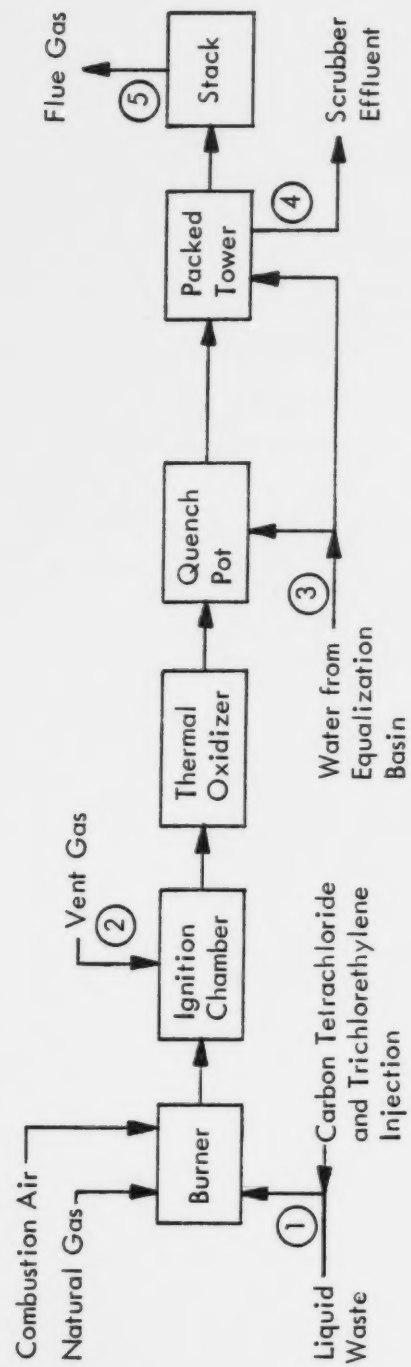
PROCESS DESCRIPTION AND OPERATING DATA

The incinerator tested is a facility located in a chemical plant that is designed to burn gaseous and liquid production wastes. It consists of a small burner/ignition chamber section, followed by a horizontal, cylindrical thermal oxidizer chamber. Combustion gases leave the oxidizer and travel downward through a spray chamber and quench pot before entering a vertical scrubber/demister and exiting via the exhaust stack. A schematic diagram of the system is shown in Figure E-1.

Liquid waste is stored in two nitrogen-pressurized, cylindrical tanks. Flow is manually controlled by adjusting the tank pressure. The gaseous wastes are piped in directly from a production area and can be automatically diverted to backup scrubbers in the event of an incinerator shutdown. Natural gas is used as an auxiliary fuel to maintain proper temperature control. The liquid waste, natural gas, and combustion/atomization air are fed to the burner assembly, while waste gases are fed to the ignition chamber (5-1/2 ft outside diameter by 8 ft long). The combustion chamber is 20 ft long with an outside diameter of about 8 ft. The unit is rated at 15×10^6 Btu/hr. Typical operating temperature is around 2000°F, with a retention time of 3 to 4 sec. A drawing of the combustion chamber is presented in Figure E-2.

The combustion chamber is connected to the quench pot by a vertical tube containing four spray cooling nozzles (~ 100 gpm of water). The tube extends downward into the quench pot and terminates just above the normal water level. Gases leaving the quench pot are cooled to about 170°F and travel horizontally to the wet scrubber. The scrubber is a single pass, counter-current packed tower with an integral, mesh-type demister and a self-supporting exhaust stack. Water is supplied to the scrubber system from a water treatment plant, and the effluent is returned to a nearby caustic retention pond. Input flow rate to the scrubber is usually about 200 gpm.

System operating data obtained during the tests are summarized in Table E-3 for Runs 2, 3, and 4. Run 1 was aborted due to sampling problems. The data show that the unit was operated at less than half of its rated capacity (6 MMBtuh versus 15 MMBtuh), but within the normal operating range. Waste feed flow to the incinerator is dependent upon the production rates of other portions of the chemical plant. All other parameters appeared normal and steady, except for minor fluctuations in the fuel gas and vent gas feed rates. Also, the calculated residence time (5.2 sec) was slightly greater than the stated design residence time of 3 to 4 sec.



NOTE:
Fugitive emission sampling was also carried out.

Figure E-1. Schematic diagram of process and sampling points.

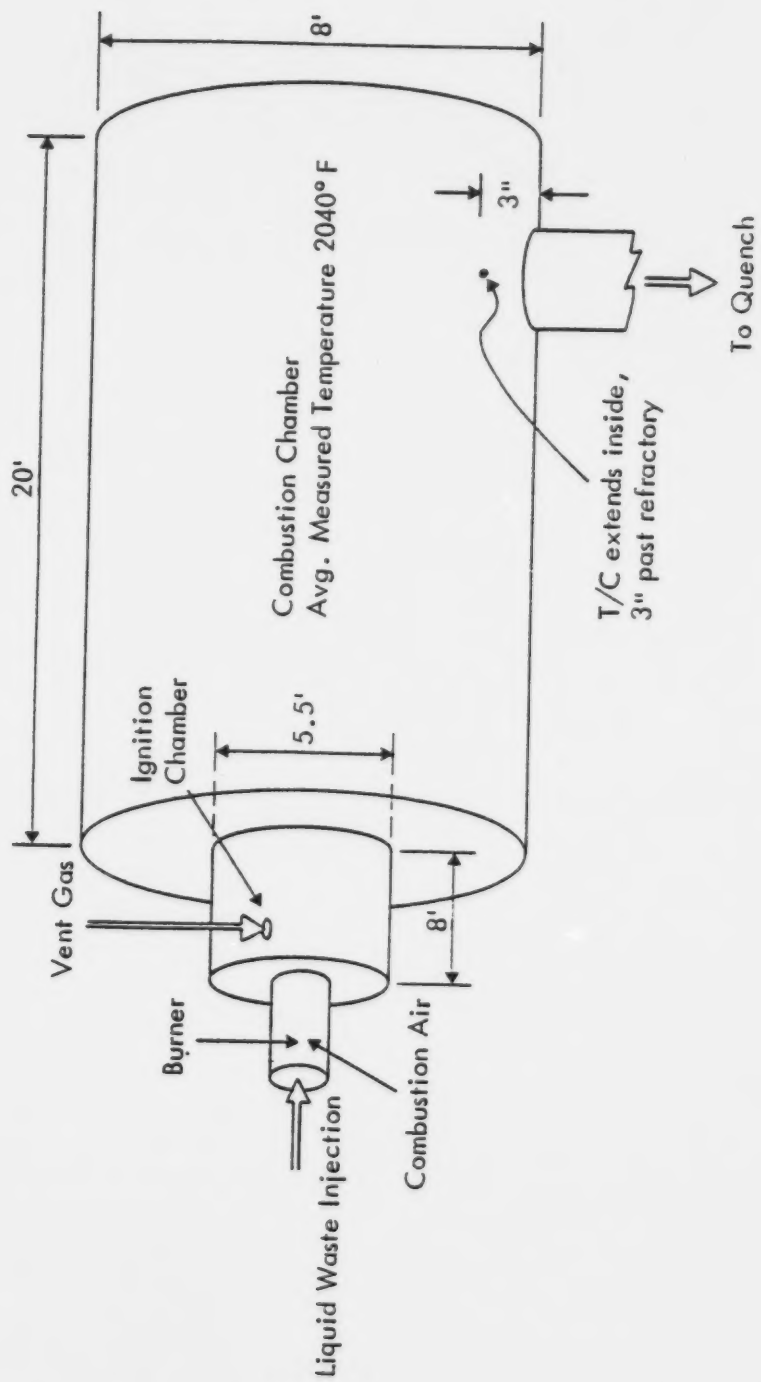


Figure E-2. Combustion chamber diagram.

TABLE E-3. INCINERATOR OPERATING DATA

	Run 2 8/12/82	Run 3 8/13/82	Run 4 8/13/82
Liquid waste flow rate - kg/min (lb/hr)	2.21 (293)	1.84 (243)	1.84 (243)
Vent gas flow rate - scmm (scfm)	7.4 (262)	7.9 (278)	7.7 (272)
Natural gas flow rate - scmm (scfm)	0.63 (22.25)	0.86 (30.5)	0.80 (28.25)
Heat input - GJ/hr (MMBtu/hr)	6.2 (5.9)	6.3 (6.0)	6.1 (5.8)
Combustion chamber temp. - °C (°F)	1116 (2040)	1116 (2040)	1116 (2040)
Calculated residence time - sec	5.2	5.2	5.2
Combustion chamber % O ₂	8.1	8.3	8.4
Cooling spray water flow rate - m ³ /min (gal/min)	0.48 (127)	0.48 (127)	0.48 (127)
Quench pot temp. - °C (°F)	84 (183)	85 (185)	85 (185)
Scrubber water flow rate - m ³ /min (gal/min)	0.61 (160)	0.61 (160)	0.61 (160)

SECTION 4

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

Table E-4 presents a summary of the sampling and analysis activities conducted for this particular test site and is organized according to the sampling points previously identified in Figure E-1. In addition to the comprehensive sampling of the stack effluent, samples of the two waste feeds and the input and effluent liquids for the quench and scrubber were also collected.

Liquid waste samples were easily obtained using an existing valve in the line between the storage tank and the incinerator. Three grab samples were collected and composited for each sampling run. Collecting samples of the waste vent gas proved to be a more difficult task due to its physical state and the hazardous nature of its components. Three sampling methods were used to characterize the major components. Total chloride content was determined by collection in an evacuated flask containing a dilute sodium hydroxide solution and subsequent analysis using an autoanalyzer (EPA Method 325.2). Phosgene content was determined by withdrawing 10 cc of the waste gas from a sample line via an in-line tee connection containing a septum. The gas-tight syringe was then returned to the field laboratory and the sample injected into a closed vessel containing an absorbing reagent. Analysis was done colorimetrically. Additional samples of the waste vent gas were collected in double-ended glass sampling vessels and were returned to the analytical laboratory for laboratory analysis by GC/MS for POHCs and other major constituents.

A 50:50 mixture (by volume) of carbon tetrachloride (CCl_4) and trichloroethylene (TCE) was continuously pumped (spiked) into the liquid waste feed line at a rate of about 100 mL/min. The valve used for collecting the liquid waste samples was about 10 ft downstream of the spiking location, and both locations were at least 35 ft from the incinerator burner unit.

Grab sampling techniques were used to obtain samples of the quench spray/scrubber water. Input samples were collected from a valve just upstream from the quench spray section, and the outlet samples were collected from the sump at the base of the scrubber tower. One grab sample of inlet water was taken per run, while a composite of three samples was collected from the sump during each run.

Stack sampling methods included a modified EPA Method 5 for particulates, semivolatile POHCs and PICs, and chlorides; two methods for collecting volatile POHCs and PICs, an integrated gas bag and the volatile organic sampling train (VOST); two midjet impinger trains operated in parallel for

TABLE E-4. SUMMARY OF SAMPLING AND ANALYSIS ACTIVITIES

Sampling point	No. of samples per run	Sampling technique	Analysis
<u>Waste feeds</u>			
1. Liquid waste	1 composite	3 grab samples	Ash, POHCs, Cl ⁻ , HHV, viscosity Cl ⁻ COCl ₂
2. Vent gas	2 grabs 2 grabs	Evacuated flask with reagent Syringe (collect 10 cc and inject into reagent)	
	2 grabs	Glass gas cylinder	POHCs
<u>Scrubber liquids</u>			
3. Input to quench and scrubber	1 grab	Grab sample	Cl ⁻
4. Combined scrubber/ quench effluent	1 composite	3 grab samples	Cl ⁻
<u>Combustion effluent</u>			
5. Stack	1	Modified EPA Method 5	Particulate weight, semivolatile POHCs and PICs, Cl ⁻ Volatile POHCs and PICs
	1	VOST	Volatile POHCs and PICs
	1	Integrated gas bag	Volatile POHCs and PICs
	1	Midget impinger train (parallel sets of impingers)	COCl ₂ , isocyanates
	1	Integrated gas bag	Orsat - CO ₂ , O ₂
	Continuous	Continuous gas analyzers	CO, O ₂ , CO ₂ , THC
	Intermittent	Certified observer	Opacity

phosgene and isocyanates (analyzed on-site colorimetrically); an integrated gas bag for Orsat determination of CO₂ and O₂; and continuous gas analyzers for O₂, CO, CO₂, and total hydrocarbons. A schematic diagram of the MM5 train is shown in Figure E-3, and the VOST is diagrammed in Figure E-4.

Gas bag samples were collected in 15-L Tedlar bags, drawing gas into the bags over a period of about 1 hr. The VOST train consists of a pair of Tenax and Tenax/charcoal traps (one each). Gas is drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The pair of traps is then removed and replaced with another pair, until a total of six pairs have been used (i.e., 120 min of sampling time). These traps and the gas bags are then returned to the analytical laboratory for analysis of volatile POHCs.

Replicates of the liquid waste feed samples were collected for determination of higher heating value and chloride content. The remaining waste feed analyses for POHCs were done using GC/MS techniques. Prior to analysis, the samples were homogenized either manually or mechanically depending upon their physical state. The homogenized feed samples were analyzed for volatile POHCs using a method based on Method A101b in the A. D. Little report entitled "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0 g aliquot of the mixed waste feed sample was dispersed in 40 mL of prepurified tetraglyme. A 10- μ L aliquot of the tetraglyme solution was spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample was then analyzed by the purge-trap-desorb technique as described in Method A101A in the A. D. Little report (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis may be repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs were analyzed in the waste feed samples according to Method P023 in the A. D. Little report. A 5-g portion of the homogenized waste feed sample was spiked with semivolatile surrogate compounds, such as decafluorobiphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 μ g/g depending on the expected sample complexity. The sample was allowed to equilibrate with the spiked compounds and then was extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appeared on the surface of the methylene chloride, the pH was checked and the sample pH neutralized. The extract was then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

An aliquot of the extract was then screened by gas chromatography with a flame ionization detector. If necessary, the extract was further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column (FSCC). A 1.0 mL aliquot of the extract was then spiked with 20 μ g of D₁₀-anthracene internal standard and analyzed by FSCC GC/MS.

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with the VOST. A known amount of the gas bag sample, usually 5.0 L, was transferred onto

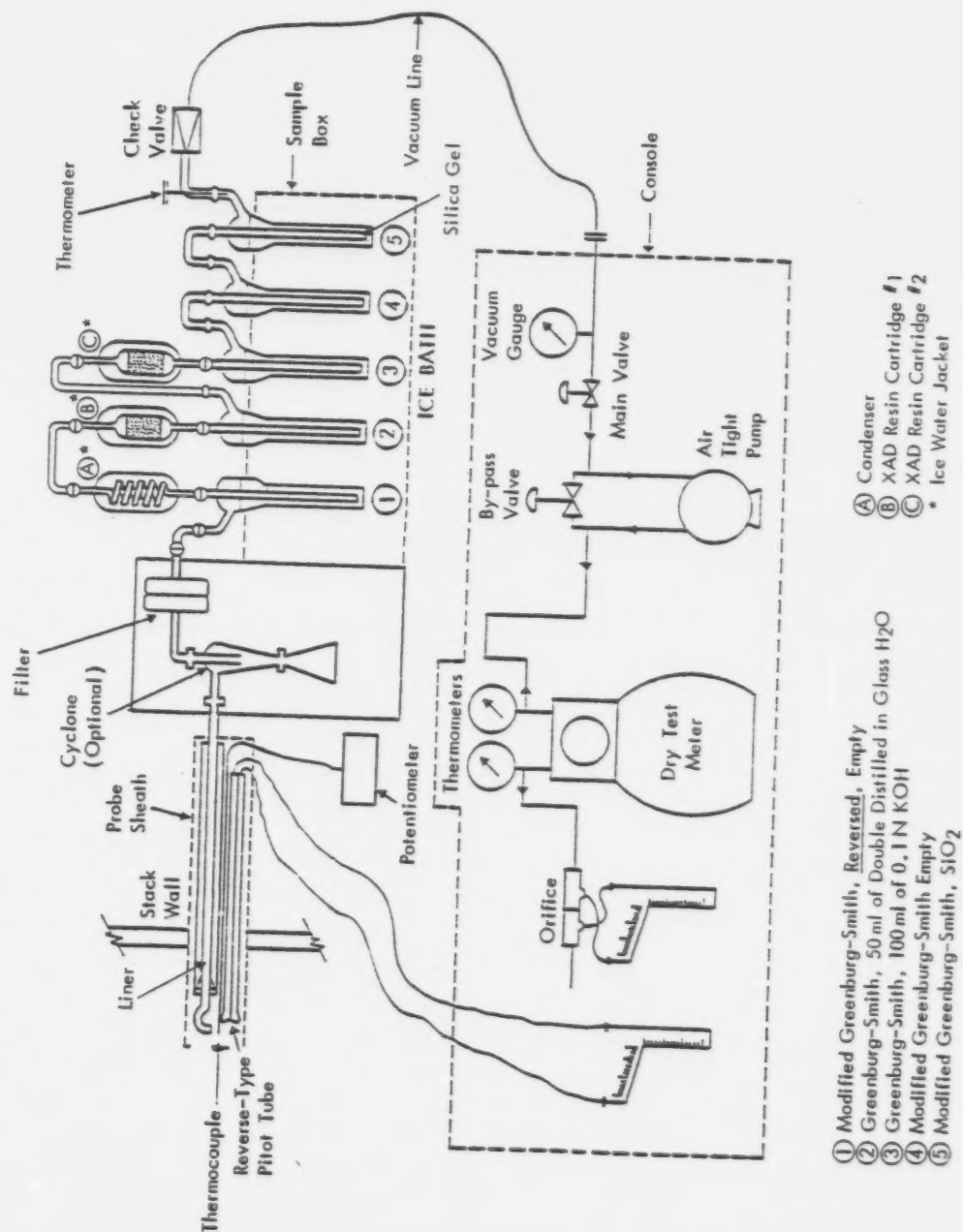
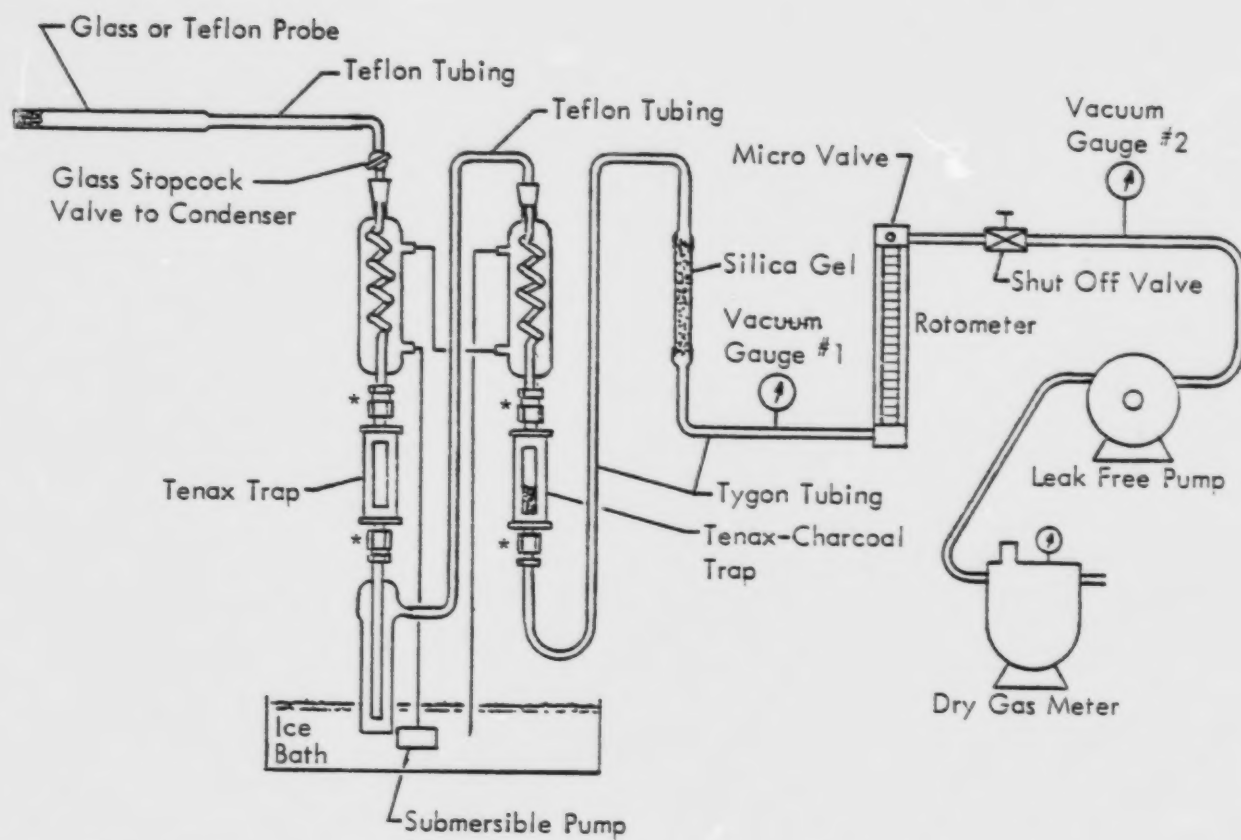


Figure E-3. Modified Method 5 sampling train (MM5).



* Special O-Ringed Stainless Steel Fittings

Figure E-4. Volatile organic sampling train (VOST).

a Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). A needle was inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were analyzed using the same techniques as for the VOST traps described below.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene (BFB) internal standards using the flash vaporization technique in which the spiking solution was vaporized and carried onto the trap with a carrier gas. The glass traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, was turned on through the trap and a syringe needle injected through the GC septum port. One microliter of a 25 ng/μL methanolic solution of the internal standards was slowly expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL) which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds passed into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs in the stack samples involves several components of the MM5 train. Each of the components of the MM5 train were spiked with a separate surrogate standard according to the following scheme:

Front XAD-2 resin trap: 20 μg of ¹³C-tetrachlorobiphenyl

Back XAD-2 resin trap: 20 μg of decafluorobiphenyl

Particulate filter plus probe rinse residue: 20 μg of D₁₂-chrysene

Condensate water: 20 μg of D₈-naphthalene

The spiked components were normally extracted separately, and then the extracts were combined, dried with anhydrous sodium sulfate, concentrated to 1.0 mL using Kuderna-Danish evaporation according to Method P031 in the A. D. Little report and using supplementary gentle nitrogen blowdown, and analyzed by FSCC GC/MS. The samples were extracted according to the following procedures:

XAD-2 resin traps: The XAD-2 resin traps were Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Particulate filters plus probe rinse residues: The residue from the acetone rinse of the probe was added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Condensate water: The condensate water was extracted with three 60-mL aliquots of methylene chloride as described in Method P021a in the A. D. Little report (equivalent to EPA Method 625).

Analysis for POHCs in scrubber water samples was similar to the procedures described above. Particulate and chloride analyses were conducted according to EPA Method 5 and EPA Method 325.2 from "Methods for Chemical Analysis of Water and Wastes," respectively.

Chlorine content was determined by potentiometric titration using a Fisher Titralyzer Model 41. Heating value was determined according to ASTM Methods D-240-64 and D-2382.

EPA Method 3 was followed when conducting Orsat analysis of the stack effluent. The continuous monitoring instrumentation consisted of the following:

- O₂ - Beckman Model 742 (polarographic sensor)
- CO - Beckman Model 215A (nondispersive infrared)
- HC - Beckman Model 402 (heated oven/flame ionization detector)
- CO₂ - Horiba Model PIR-2000S (nondispersive infrared)

More detailed descriptions of all the above sampling and analysis methods are presented in Appendix B.

SECTION 5

PRESENTATION OF RESULTS

INPUT AND OUTPUT CONCENTRATIONS AND RATES FOR POHCs

POHC input and output concentrations and mass rates were determined for all POHCs listed by EPA which were detected in the waste feeds at concentrations above 100 $\mu\text{g/g}$. No attempt was made to restrict the number of POHCs based on any selection or ranking method. The POHCs reported were quantified against authentic standards whenever possible. Where authentic standards were not available, estimated GC/MS response factors were used to provide semiquantified results.

The concentrations of both volatile and semivolatile POHCs measured in the two waste feed streams are shown in Table E-5. The same results, expressed as input rates (g/min), are presented in Table E-6 and were based on the waste feed rates previously presented in Table E-3.

Table E-7 summarizes volatile POHC concentrations and emissions rates. Table E-8 contains the same information for semivolatile compounds.

Semivolatile POHC results were fairly consistent among the three sampling runs, although the values reported for all of the semivolatiles in Run 3 were greater than the results of the other two runs. Neither aniline nor phenyl isocyanate could be detected in the modified Method 5 samples (both were only semiquantifiable because of the lack of standard reference materials). Other forms of isocyanates could not be defined by the Method 5 sampling-GC/MS analysis method. A separate sampling and analysis method was utilized, however, to measure phenyl isocyanates and the results are reported later in this section.

EMISSION OF PICs

The stack effluent sample analysis included the identification of PICs, i.e., those compounds identified in the emission samples that are listed as POHCs by EPA, but which were not measured in the waste feed samples at concentrations above 100 $\mu\text{g/g}$. The emission concentrations and output rates for volatile PICs are tabulated in Table E-9. The most significant PIC appeared to be chloroform, which was found in concentrations ranging from the 280 to 2,800 ng/L . To a lesser degree, but still relatively significant, were the concentrations of benzene measured. Many of the PICs exhibited a good deal of variability from run to run, which one might expect given the nature of their production (i.e., fluctuating combustion conditions). Semivolatile PIC results are shown in Table E-10.

TABLE E-5. UPJOHN CONCENTRATIONS OF POHCs IN WASTE FEEDS

	Liquid waste ($\mu\text{g/g}$)		Vent gas ($\mu\text{g/L}$)	
	Run 2	Run 3	Run 2	Run 3
Volatiles POHCs				
Carbon tetrachloride	36,000 ^g	44,000 ^g	2.0	5.7
Trichloroethylene	33,000 ^g	40,000 ^g	0.10	0.045
Chlorobenzene ^f	7,200	4,100	< 0.005	< 0.005
Chloromethane	> 2,000	> 1,200	< 0.005	< 0.005
Semivolatiles POHCs				
m-Dichlorobenzene	2,100	2,300	c	c
o-Dichlorobenzene	40,000	46,000	c	c
p-Dichlorobenzene	56,000	59,000	c	c
1,2,4-Trichlorobenzene	270	290	c	c
Bis(ethylhexyl)phthalate	500	500	c	c
Phenyl isocyanate	170,000	160,000	c	c
Chlorophenyl isocyanate	23,000	21,000	c	c
Aniline ^e	a	14,000	c	c
Phosgene ^e	b	b	534,000 ^d	508,000 ^d
				202,000 ^d

a Result not determinable due to interferences.

b Highly unlikely as a waste constituent, therefore, not analyzed in sample.

c Vent gas samples not analyzed for semivolatiles.

d Separate sampling and analysis conducted for phosgene.

e Phosgene does not appear on EPA's Appendix VIII listing of organic hazardous constituents, but it is the major constituent of the vent gas waste stream and is a hazardous chemical.

f Semiquantified based on common relative response to D₆ benzene internal standard.

g These concentrations based on spiking rates.

h The presence of aniline in the waste feed is probably due to the hydrolysis of phenylisocyanates.

TABLE E-6. CALCULATED INPUT RATES FOR POHCs IN WASTE FEEDS

	Input rates (g/min)		
	Run 2	Run 3	Run 4
<u>Volatile POHCs</u>			
Carbon tetrachloride	80	85	84
Trichloroethylene	73	74	74
Chlorobenzene	16	7.5	13
Chloromethane	> 4.4	> 2.2	> 3.5
<u>Semivolatile POHCs</u>			
m-Dichlorobenzene	4.6	4.2	5.7
o-Dichlorobenzene	89	85	120
p-Dichlorobenzene	120	110	150
1,2,4-Trichlorobenzene	0.60	0.53	0.72
Bis(ethylhexyl)phthalate	1.1	0.92	2.4
Phenyl isocyanate	380	290	390
Chlorophenyl isocyanate	51	39	51
Aniline	-	26	35
Phosgene	<u>3,950</u>	<u>4,010</u>	<u>1,560</u>
Total	> 4,700	> 4,640	> 2,410

TABLE E-7. EMISSIONS OF VOLATILE POHCs (NOT BLANK CORRECTED)

	Gas bags				VOST			
	Run No. 2	Run No. 3	Run No. 4	Run No. 2	Run No. 3	Run No. 4	Run No. 2	Run No. 3
	1st Pair	4th Pair	6th Pair	1st Pair	4th Pair	6th Pair	1st Pair	4th Pair
	Concentration (ng/L)						6th Pair	
Carbon tetrachloride ^a	92	110	72	110	110	88	97	110
Trichloroethylene ^a	32	24	6	310	34	230	39	30
Chlorobenzene ^b	c	200	140	260	240	150	143	110
Chloromethane	1.2	2.0	1.6	12	c	6.1	3.4	11
							4.9	3.6
	Output rate (g/min)						6th Pair	
Carbon tetrachloride ^a	0.0049	0.00058	0.0039	0.0056	0.0061	0.0074	0.0052	0.0059
Trichloroethylene ^a	0.0017	0.0013	0.00032	0.016	0.0018	0.011	0.012	0.0016
Chlorobenzene ^b	c	0.011	0.0076	0.014	0.013	0.0071	0.0080	0.0014
Chloromethane	0.000064	0.00011	0.000086	0.00066	c	0.00032	0.00020	0.00054
							0.00026	0.00059
								0.00020

^a Quantified relative to authentic calibration standard.^b Quantified relative to perfluorobenzene standard.^c No data for this compound because of analytical interferences.

TABLE E-8. EMISSIONS OF SEMIVOLATILE POHCs (NOT BLANK CORRECTED)

	Concentration (ng/L)		
	Run 2	Run 3	Run 3
m-Dichlorobenzene ^a	67	75	72
o-Dichlorobenzene ^a	12	110	24
p-Dichlorobenzene	22	100	8.5
1,2,4-Trichlorobenzene ^a	40	140	33
Bis(ethylhexyl)phthalate ^a	4.7	9.2	7.8
Phenyl isocyanate ^b	< 5	< 5	< 5
Aniline ^b	< 5	< 5	< 5
	Output rate (g/min)		
	Run 2	Run 3	Run 3
m-Dichlorobenzene ^a	0.0036	0.0040	0.0039
o-Dichlorobenzene ^a	0.00063	0.0054	0.00046
p-Dichlorobenzene ^a	0.0012	0.0058	0.0013
1,2,4-Trichlorobenzene ^a	0.0021	0.0074	0.0018
Bis(ethylhexyl)phthalate ^a	0.00025	0.00049	0.00042
Phenyl isocyanate ^b	< 0.0003	< 0.0003	< 0.0003
Aniline ^b	< 0.0003	< 0.0003	< 0.0003

^a Quantified relative to authentic calibration standard.

^b Semiquantified based on an estimated GC/MS response factor.

TABLE E-9. EMISSIONS OF VOLATILE PICs (NOT BLANK CORRECTED)

Compounds	Gas Bags			VOST					
	Run 2	Run 3	Run 4	Run 2		Run 3		Run 4	
	1st pair	4th pair	6th pair	1st pair	4th pair	1st pair	4th pair	1st pair	4th pair
Concentration (ng/L)									
Chloroform ^a	2,800	640	320	5,100	800	5,000	430	270	250
Benzene ^a	52	220	36	66	27	30	110	140	36
Tetrachloroethylene ^a	5.4	2.8	1.8	3.2	1.3	2.7	3.0	6.2	2.0
Toluene ^a	38	13	6.8	78	88	99	36	85	17
Methylene chloride ^b	24	22	42	16	c	19	5.6	9.7	25
Methyl ethyl ketone ^b	5.8	1.8	3.8	1.3	0.88	1.4	3.3	2.7	7.6
Bromodichloromethane ^b	260	44	14	87	82	54	31	21	20
Dibromochloromethane ^b	32	3	1.2	48	37	34	15	13	7.9
Output rate (g/min)									
Chloroform ^a	0.15	0.034	0.017	0.27	0.042	0.26	0.023	0.014	0.019
Benzene ^a	0.0028	0.012	0.0019	0.0035	0.0014	0.0016	0.0058	0.0075	0.0020
Tetrachloroethylene ^a	0.00029	0.00015	0.000097	0.00017	0.000071	0.00014	0.00016	0.00033	0.00011
Toluene ^a	0.0020	0.00069	0.00037	0.0042	0.0047	0.0052	0.0019	0.0046	0.00091
Methylene chloride ^b	0.0013	0.0012	0.0023	0.00088	c	0.00098	0.00030	0.00080	0.0013
Methyl ethyl ketone ^b	0.00031	0.000095	0.00021	0.000071	0.000047	0.000074	0.00017	0.00015	0.00041
Bromodichloromethane ^b	0.014	0.0023	0.00077	0.0046	0.0043	0.0029	0.0017	0.0011	0.0011
Dibromochloromethane ^b	0.0017	0.00016	0.000065	0.0025	0.0020	0.0018	0.00079	0.00070	0.00043

^a Quantified relative to authentic calibration standard.^b Semiquantified relative to perfluorobenzene internal standard.^c No data for this compound due to analytical interference.

TABLE E-10. EMISSIONS OF SEMIVOLATILE PICs
(NOT BLANK CORRECTED)

Compound	Run 2	Run 3	Run 4
Concentration (ng/L)			
Phenol	9.1	3.1	< 0.7
Naphthalene	1.3	7.2	6.5
2,6-Toluene diisocyanate	< 3	3.8	< 3
Diethyl phthalate	9.4	6.8	5.1
Hexachlorobenzene	0.61	< 0.3	0.48
o-Chlorophenol	3.0	22	1.4
2,4,6-Trichlorophenol	94	126	109
Pentachlorophenol	8.4	5.5	5.1
o-Nitrophenol	10	44	22
Output rate (g/min)			
Phenol	0.00048	0.00016	< 0.00004
Naphthalene	0.000069	0.00038	0.00035
2,6-Toluene diisocyanate	< 0.0002	0.00020	< 0.0002
Diethyl phthalate	0.00050	0.00036	0.00028
Hexachlorobenzene	0.000032	< 0.00002	0.000016
o-Chlorophenol	0.00016	0.0012	0.000076
2,4,6-Trichlorophenol	0.0050	0.0067	0.0059
Pentachlorophenol	0.00045	0.00029	0.00028
o-Nitrophenol	0.00053	0.0023	0.0012

2,4,6-Trinitrophenol and o-nitrophenol were the most prevalent semivolatile PICs. All others were less than 10 ng/L.

BLANK VALUES FOR POHCs AND PICs

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table E-11. The sample volumes for gas bags and VOST are presented in Table E-12. Blank correction data for semivolatile POHCs and PICs are presented in Table E-13. Sample volumes for the MM5 train can be found in Table E-17.

DREs FOR VOLATILE AND SEMIVOLATILE POHCs

Destruction and removal efficiencies (DREs) based on the above data are presented in Table E-14. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$\text{DRE} = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

POHCs AND CHLORIDE IN SCRUBBER WATER STREAMS

The scrubber makeup water samples were analyzed for the POHCs that were identified in waste feed samples. Results are shown in Table E-15. Only chlorobenzene and bis(ethylhexyl)phthalate were found in the samples at detectable levels.

Chloride analysis results for the scrubber makeup and effluent water samples are presented in Table E-16. The chloride concentrations both entering and leaving the packed tower scrubber were consistent among the three sampling runs. The scrubber effluent showed an increase of chloride content of slightly less than 3 mg/mL.

STACK EFFLUENT MEASUREMENTS

Particulate Emissions

Particulate emission results from the modified EPA Method 5 tests are shown in Table E-17. Other significant test parameters measured during the MM5 runs are also included for comparison. Most parameters were quite consistent among the three runs. However, the filterable particulate catch from Run 4 was inexplicably low compared to the other two runs, which were in close agreement. A recheck of the filter weight indicated that it was a true value and led to an examination of the condensate catch from the run. The condensate sample contained a visible amount of particulate material which settled out upon standing during storage (unlike the other condensate samples). An attempt was then made to evaporate the condensate to obtain a particulate weight which could be added to the filter weight.

TABLE E-11. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank values (ng)	Standard deviation (ng)	Average blank values (ng)	Standard deviation (ng)
<u>POHCs</u>				
Carbon tetrachloride	5.2	10	26	19
Trichloroethylene	42	21	170	120
Chlorobenzene	40	49	260	320
Chloromethane	< 1	0	< 1	0
<u>PICs</u>				
Chloroform	< 1	0	21	12
Benzene	18	8.8	96	25
Tetrachloroethylene	6.8	3.9	22	9.3
Toluene	52	19	730	510
Methylene chloride	14	4.0	320	450
Methyl ethyl ketone	1	0	5.9	4.1
Bromodichloromethane	< 1	0	< 1	0
Dibromochloromethane	< 1	0	< 1	0

TABLE E-12. VOLATILE SAMPLE VOLUMES (DRY STANDARD LITERS)

Gas bags		VOST	
Run No.	Sample volume (L)	Run No./Pair No.	Sample volume (L)
2	5.0	2/1	18.7
3	5.0	2/4	19.3
4	5.0	2/6	19.5
		3/1	18.8
		3/6	19.5
		4/1	20.0
		4/4	19.1
		4/6	19.2

TABLE E-13. SEMIVOLATILE BLANK CORRECTION VALUES

Compound	Blank correction value (μg)
<u>POHCs</u>	
m-Dichlorobenzene	< 1
o-Dichlorobenzene	< 1
p-Dichlorobenzene	< 1
1,2,4-Trichlorobenzene	< 1
Bis (ethyl hexyl) phthalate	1.4
Phenyl isocyanate	< 5
Aniline	< 5
<u>PICs</u>	
Phenol	< 1
Naphthalene	7.8
2,6-Toluene diisocyanate	< 5
Diethyl phthalate	1.5
Hexachlorobenzene	< 5
o-Chlorophenol	< 2
2,4,6-Trichlorophenol	< 2
Pentachlorophenol	< 5
o-Nitrophenol	< 5

TABLE E-14. DESTRUCTION AND REMOVAL EFFICIENCIES (DRES) FOR THE UPJOHN INCINERATOR (BLANK CORRECTED)

Volatile compounds	Gas bags				Fast VOST	
	Run 2	Run 3	Run 4	Run 2	Run 3	Run 4
Carbon tetrachloride	99.9940	99.9931	99.9954	99.9925	99.971	99.988
Trichloroethylene	99.9983	99.9989	> 99.9956	> 99.9922	99.9914	99.9914
Chlorobenzene	IF	99.86	99.945	99.937	99.910	99.956
Chloromethane	> 99.9986	> 99.9952	> 99.9975	> 99.990	> 99.9916	> 99.9903
Semivolatiles compounds						
NM5						
Run 2 Run 3 Run 4						
m-Dichlorobenzene	99.922	99.905	99.932			
o-Dichlorobenzene	99.9990	99.993	99.9990			
p-Dichlorobenzene	99.9990	99.995	99.9990			
1,2,4-Trichlorobenzene	99.65	98.6	99.75			
Bis(ethylhexyl)phthalate	99.98	99.95	99.98			
Phenyl isocyanate	> 99.99992	> 99.99990	> 99.99992			
Aniline	^a	> 99.9988	> 99.9991			
Phosgene	99.9985	99.9930	99.981			

IF Quantification of this compound prohibited by interfering peak on GC/MS.

^a Waste feed concentration < 100 µg/g in this run.

TABLE E-15. CONCENTRATION OF POHCs IN SCRUBBER
MAKEUP WATER ($\mu\text{g/L}$)

	Run 2	Run 3	Run 4
<u>Volatile POHCs</u>			
Carbon tetrachloride	ND ^a	ND	ND
Trichloroethylene	ND ^a	ND	ND
Chlorobenzene	> 200	40	46
Chloromethane	ND ^a	ND	ND
<u>Semivolatile POHCs</u>			
m-Dichlorobenzene ^c	ND ^b	ND	ND
o-Dichlorobenzene ^c	ND ^b	ND	ND
p-Dichlorobenzene ^c	ND ^b	ND	ND
1,2,4-Trichlorobenzene ^c	ND ^b	ND	ND
Bis(ethylhexyl)phthalate ^c	23	87	68
Phenyl isocyanate ^d	ND ^b	ND	ND
Aniline ^d	ND ^b	ND	ND

- ^a ND = None detected; detection limit is 10 $\mu\text{g/L}$.
- ^b ND = None detected; detection limit is 1 $\mu\text{g/L}$.
- ^c Quantified by comparison with authentic standard and utilizing internal standard.
- ^d Semiquantified using an estimated GC/MS response factor.

TABLE E-16. CHLORIDE CONTENT OF SCRUBBER MAKEUP AND EFFLUENT WATERS (mg/mL)

	Run 2	Run 3	Run 4
Makeup concentration	20.6	21.9	20.9
Effluent concentration	23.4	24.6	23.8
Difference	2.8	2.7	2.9

TABLE E-17. MODIFIED METHOD 5 TEST DATA

	Run 2	Run 3	Run 4
Volume of gas sampled (Nm ³ , dry)	2.964	2.928	2.930
Sampling time (min)	180	180	180
Percent isokinetic	101.2	100.1	99.7
Moisture content (%)	13.1	10.4	13.2
Percent O ₂ (dry)	8.4	8.2	8.1
Percent CO ₂ (dry)	13.8	13.9	14.4
Stack flow rate (actual m ³ /min)	68	65	68
Stack temperature (°C)	51	47	52
Stack flow rate (Nm ³ /min, dry)	53	53	54
Particulate concentration			
(mg/dscm)	195.7	167.0	26.5 ^a
(gr/dscf)	0.0853	0.0728	0.0116 ^a
(mg/dscm corr. to 7% O ₂)	217.4	182.7	28.8 ^a
(gr/dscf corr. to 7% O ₂)	0.0948	0.0796	0.0126 ^a

^a Filter weight was very low for this run. A noticeable amount of particulate was found in the condensate catch, indicating that some of the particulate material was not captured by the filter. An attempt was made to evaporate the condensate sample to determine the weight of particulate residue. However, some unknown material (non-filterable) was also found to comprise the residue after evaporation and thus invalidated a determination of the filterable particulate weight.

However, evaporation of the liquid catch produced a whitish residue in addition to the dark particulate material. It is surmised that some type of inorganic salt (nonparticulate) comprised this unexpected residue. The condensate from Run 3 also produced the same material when evaporated. Therefore, a "true" particulate weight could not be determined for Run 4.

Results from Runs 2 and 3 showed that particulate emissions from the Upjohn incinerator were slightly in excess of the federal limitation (0.08 gr/scf) when corrected to 7% O₂. The particulate concentration determined from Run 3 equaled 0.08 gr/scf, while the Run 2 result was higher at 0.095 gr/scf.

Opacity

On the second day of testing, the stack plume opacity was evaluated by one of the field crew who is a certified visible emissions observer. Plume opacity could not be evaluated at other times during the test program because of overcast skies or darkness. Three sets of 6 min observations indicated that the plume opacity ranged from 16 to 19%. All 15 sec readings were between 10 and 25%.

Chlorides

A summary of chloride emissions is presented in Table E-18. Results are expressed as the concentrations measured in the MMS samples, as well as the total output rate (g/min). For comparison, the total input rates (total chlorides in the waste feeds) are also shown, and the resulting chloride removal efficiency is given. The incinerator system's removal efficiency was slightly less than the 99.9% removal rate specified by the Texas Air Control Board. However, the total emission rate is well below the federal limitation of 4 lb/hr.

Phosgene

Results of the phosgene emission measurements and comparative concentrations in the vent gas waste streams are shown in Table E-19. The levels of phosgene detected in the stack gas were approximately 1 ppm (≤ 0.30 g/min emitted).

Isocyanates

Isocyanate emission data are presented in Table E-20, along with measured levels of isocyanates in the liquid waste samples. Measurement of isocyanates in the waste input material and stack effluent gases presented a particular problem for this test program because of the varied forms of isocyanates suspected in the waste and their chemical reactivity. Analysis of the liquid waste samples was done by GC/MS, which resulted in only semi-quantitative results because authentic standard materials were not available. Also, the sample preparation and extraction procedures prior to analysis probably interfered with the detection of some isocyanate forms (such as butyl isocyanate). Upjohn data indicated that isocyanates should

TABLE E-18. CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

	Run 2	Run 3	Run 4
Total Cl ⁻ in stack effluent			
mg/dscm	132	168	239
g/min	7.0	8.9	13
lb/hr	0.93	1.2	1.7
Total Cl ⁻ in waste feed			
g/min	3,250	4,560	3,630
lb/hr	430	603	480
% Cl ⁻ removal	99.79	99.81	99.64

TABLE E-19. PHOSGENE EMISSIONS

	Run 2	Run 3	Run 4
Phosgene in stack effluent			
mg/m ³	1.1	5.2	5.5
ppm	0.27	1.3	1.4
g/min	0.058	0.28	0.30
Phosgene in vent gas waste			
mg/m ³	534,000	508,000	202,000
ppm	132,000	126,000	50,000
g/min	3,950	4,010	1,560

TABLE E-20. ISOCYANATE EMISSIONS

	Run 2	Run 3	Run 4
Isocyanates in stack effluent ^a			
mg/m ³	< 0.1	0.63	0.50
ppm	< 0.01	0.06	0.05
g/min	< 0.005	0.033	0.027
Isocyanates in liquid waste ^b			
μg/g	190,000	180,000	240,000
g/min	430	330	440

^a Values shown represent total isocyanates based on NIOSH sampling method for diphenylmethane diisocyanate.

^b Values shown represent semiquantitative evaluation of phenyl and chlorophenyl isocyanate components only. Other forms of isocyanates are suspected to be present in the waste material. Therefore, the total quantity of isocyanates is likely to be higher than indicated.

comprise about 85% of the liquid waste material. The highest concentrations measured (for Run 4) were about 240,000 $\mu\text{g/g}$ or 24% of the sample.

The sampling and analytical method used to measure isocyanates in the stack effluent was based on a NIOSH method for diphenylmethane diisocyanate (MDI) in air, but is not specific for MDI. Therefore, other forms of isocyanates could be detected by this method, although it is not likely to be a universal method. Levels of total isocyanates in the stack effluent would not be expected to be much greater than the levels measured because of the high reactivity of isocyanates with water.

Gaseous Effluents

Table E-21 presents a summary of the data obtained through continuous monitoring of the stack gases during the MMS runs. Results are given as the average value for each run, as well as the range of values recorded during each run. All of the constituents measured were very consistent during the three sampling runs.

GENERAL WASTE FEED ANALYSIS

Results of the liquid waste feed sample analysis for total chloride, ash, viscosity, and heating value are presented in Table E-22. The data indicate a consistent liquid waste material was fed to the incinerator during the test series. A determination of water content was not made due to the strong reactivity of water with the isocyanates present in this waste stream.

Table E-23 summarizes the results of the vent gas waste stream analysis for total chlorides and phosgene. The variations among the results appears to indicate that the composition of the vent gas is dependent upon fluctuations in the phosgene production process. It should also be noted that the feed rate of the vent gas from the production units to the incinerator fluctuated during the tests. The lower concentration of phosgene reported for Run 4 may have been the result of sampling error (only 10 cm^3 of gas were withdrawn from the sample line) or a momentary fluctuation in the phosgene concentration of the vent gas.

The vent gas samples were not analyzed for their heating value because of the low estimated heat content of the gas (about 100 Btu/ft^3 according to Upjohn estimates) and the complexity of performing heat of combustion analyses on gaseous samples. The Upjohn estimate is considered sufficient for the purpose of heat input calculations.

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (1) external auditing; (2) sampling QA activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

TABLE E-21. CONTINUOUS MONITORING DATA^a

Effluent constituent	Run 2	Run 3	Run 4
Oxygen (%)			
Range	8.1 - 8.7	7.2 - 8.1	7.7 - 8.1
Average	8.4	7.9	8.0
Carbon dioxide (%)			
Range	14.0 - 14.2	14.2 - 14.6	14.1 - 14.4
Average	14.1	14.4	14.3
Carbon monoxide (ppm _v)			
Range	8.0 - 11.1	9.6 - 10.3	7.8 - 9.1
Average	9.5	10.1	8.5
Total hydrocarbons (ppm _v) ^b			
Range	6.8 - 20.5	3.9 - 25.9	2.8 - 20.3
Average	8.8	5.8	3.5

^a Concentrations reported on dry gas basis.

^b Total hydrocarbons reported as propane.

TABLE E-22. GENERAL ANALYSIS OF LIQUID WASTE FEED

	Run 2	Run 3	Run 4
Heating value, kJ/kg (Btu/lb)	23,770 (10,230)	23,510 (10,110)	23,970 (10,320)
Percent chloride	21.41	22.13	21.10
Percent ash	0.17	0.19	0.21
Viscosity (sec at 100°F)	-	47.2	-

TABLE E-23. GENERAL ANALYSIS OF VENT GAS WASTE FEED

	Run 2	Run 3	Run 4
Total chloride, mg/L	376	526	421
Phosgene, mg/L (percent)	534 (13.2)	508 (12.6)	202 (5.0)

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. A comprehensive performance audit (quantitative evaluation) and limited systems audit (qualitative evaluation) were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on:

- GC/MS analysis of volatile and/or semivolatile organics; and
- Technicon Autoanalyzer analysis of chlorides.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics and semivolatile organics; however, the results for the quantification of bis(2-ethylhexyl)phthalate were erratic. This portion of the analytical program is judged to be within control limits, however.

Analyses of chloride audit samples were determined to be adequate and acceptable. This part of the analytical phase is also within control limits.

Systems audits demonstrated that laboratory analyses were done according to protocol.

Sampling QA Activities

In general, sampling QA activities are scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures included spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure used was monitoring the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX F

SUMMARY OF RESULTS FROM TESTING AT THE ZAPATA INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration regulations. Emissions data for the RIA are being provided by a sampling and analysis program. EPA selected the hazardous waste incinerator at Zapata Industries, Inc., as one facility for emission measurements.

Four test runs were conducted at this site during the period September 28 to 30, 1982. Samples of waste feeds and gaseous effluents were collected and analyzed. The following sections of this appendix present a summary of results, a brief process description, a synopsis of the test plan and sampling and analysis methods, and a discussion of the test results.

SECTION 2

SUMMARY OF RESULTS

A major part of the effort on this test program was the determination of principal organic hazardous constituents (POHCs) in the waste feed and the quantification of those compounds and products of incomplete combustion (PICs) in the outlet gas. Since no semivolatile POHCs were predicted to be present in the Zapata waste, only volatile POHCs were analyzed. Carbon tetrachloride, trichloroethylene, and chlorobenzene were spiked into the waste for test purposes. The other compounds identified as POHCs in the waste feed were toluene and methylene chloride. Compounds identified as PICs in the effluent gas were chloroform, 1,1,1-trichloroethane, benzene, and tetrachloroethylene. These results are presented along with a summary of destruction and removal efficiencies (DREs) in Section 5.

This test program also included determination of particulate emissions and HCl emissions. Results of the particulate measurements showed that the average particulate concentration (corrected to 7% O₂) for normal operation during Runs 1 to 4 was 60 mg/dscm (0.026 gr/dscf) which is less than the EPA regulation for hazardous waste incinerators of 180 mg/dscm (0.08 gr/dscf).

Chloride emission rates averaged 1.1 kg/hr (2.4 lb/hr) for Runs 1 to 4 which is less than the 1.8 kg/hr (4.0 lb/hr) level set by the EPA. Part of the Cl₂ emitted was related to the compounds spiked into the waste feed for test purposes.

SECTION 3

PROCESS DESCRIPTION AND PROCESS DATA

The incinerator is an industrial facility located in North Carolina. The unit includes a pyrolytic chamber and a thermal reactor. There is no control device for particulate or HCl control (see Figure F-1).

The incinerator is normally operated for 4 to 8 hr per day, 3 days each week. To initiate incinerator operation, about three wooden pallets are placed into the pyrolytic chamber. The main natural gas burner is then turned on. After the chamber reaches 315°C (600°F) this burner shuts off and waste feed is started. The pyrolytic chamber includes automatic addition of water through a spray nozzle to maintain a temperature of 650°C (1200°F). Gases from the pyrolytic chamber pass into the thermal reactor where additional air is added. Temperature at the exit of the thermal reactor is controlled to 870°C (1600°F) by adjustment of the waste feedrate to the pyrolytic chamber. Figure F-2 presents a drawing of the combustion chambers including thermocouple (T/C) locations.

All waste is fed to the incinerator from a single waste feed tank. One drum of waste is emptied into the tank approximately every 4 hr during normal incinerator operation.

Process data obtained during the tests are summarized in Table F-1, along with information needed for dispersion modeling. These data show that the average main chamber (pyrolyzer) temperature ranged from 670° to 737°C while the secondary chamber (thermal reactor) temperature was 844° to 904°C. The calculated residence time in the secondary chamber for all four test runs ranged from 0.063 to 0.069, compared to the design residence time of 0.22 sec. Average calculated heat input to the incinerator from the waste was 1.1×10^6 kJ/hr (1.2×10^6 Btu/hr). However, the heat input for Runs 1 and 2 was 1.4×10^6 kJ/hr (1.5×10^6 Btu/hr). Lower heat inputs calculated for Runs 3 and 4 are probably not representative since the waste feed samples are not believed to be representative, as is discussed later.

The incinerator was operated normally during all four test runs. However, some of the sampling probes were moved to a sampling port higher in the stack after Run 1 (this will be discussed in more detail in Section 4).

Table F-2 lists the waste feed rates during the four test runs.

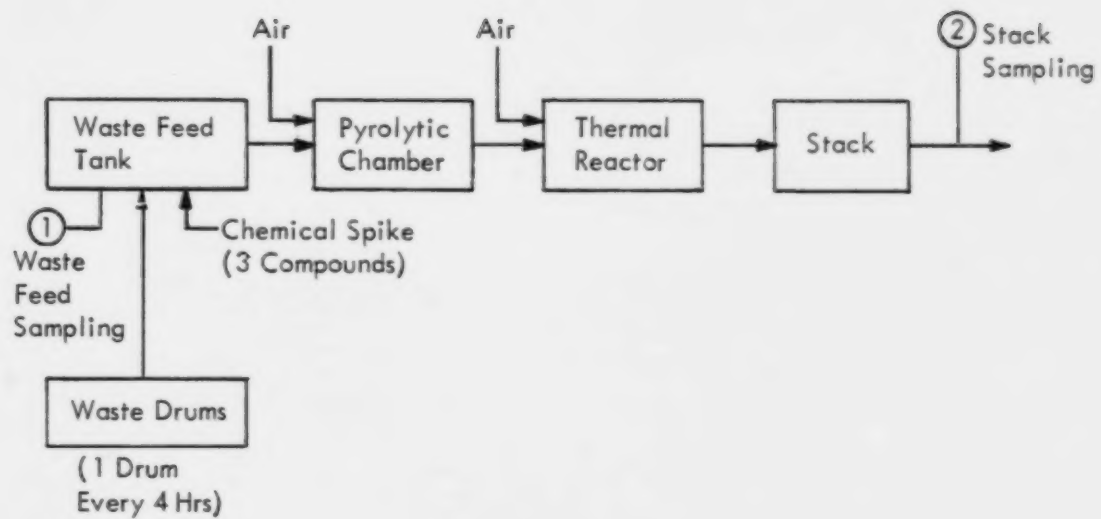


Figure F-1. Summary of sampling locations.

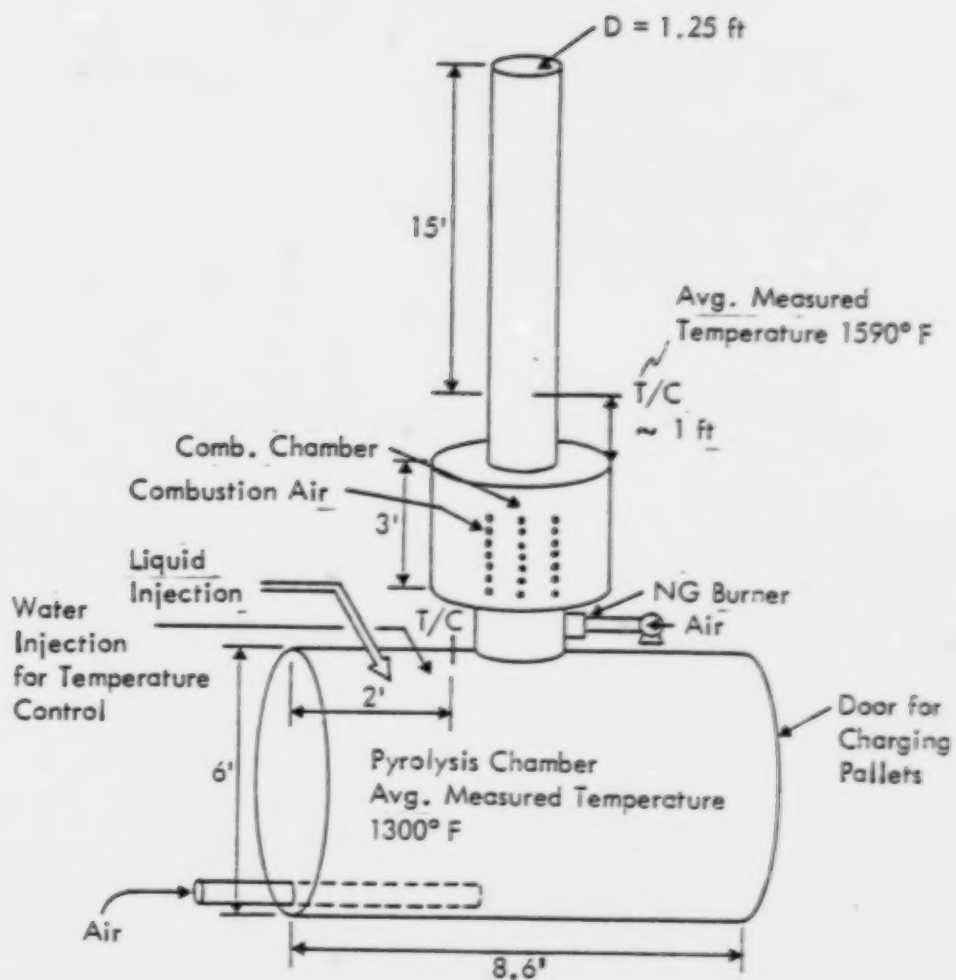


Figure F-2. Combustion chamber diagram.

TABLE F-1. PROCESS DATA

	Run 1, 9/28/82	Run 2, 9/29/82	Run 3, 9/29/82	Run 4, 9/30/82
Calculated residence time, sec	0.069	0.067	0.066	0.063
Main chamber temperature, °C (°F)	670 (1,240)	720 (1,330)	737 (1,360)	689 (1,274)
Secondary chamber temperature, °C (°F)	854 (1,570)	867 (1,594)	844 (1,553)	904 (1,661)
Heat input without natural gas, 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	1.5 (1.4)	1.7 (1.6)	1.1 ^d (1.0)	0.71 ^d (0.67)
Heat input including natural gas, 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	1.9 (1.8)	2.1 (2.0)	1.5 (1.4)	1.1 (1.0)
Main chamber pressure negative Pa (negative in H ₂ O)	19.9 (0.08)	19.9 (0.08)	17.4 (0.07)	17.4 (0.07)
Atomizing air pressure kPa (psig)	207 (30)	207 (30)	207 (30)	207 (30)
Water flow to main chamber, L/hr (gph)	18.9 (5)	41.6 (11)	45.4 (12)	41.6 (11)
Natural gas, Nm ³ /hr (scf/hr)	10.9 (385)	9.9 (350)	10.6 (375)	10.2 (360)
Electric power (kW) ^c	4	4	6.9	4
Stack exit velocity, m/min (ft/min)	656 (2,153)	637 (2,090)	687 (2,255)	702 (2,302)
Stack temperature, °C (°F)	663 (1,225)	611 (1,132)	646 (1,195)	679 (1,254)
Stack height, m (ft)	8.5 (28)	8.5 (28)	8.5 (28)	8.5 (28)

^a Residence time of secondary chamber. The design residence time is 0.22 sec.

^b Heat input from waste, not including natural gas.

^c Total electric power for fans and electrical controls, etc.

^d Calculated heat input may be low due to nonrepresentative waste feed samples.

TABLE F-2. WASTE FEED RATES

	kg/min	lb/hr
Run 1	0.66	87
Run 2	0.76	101
Run 3	0.78	103
Run 4	0.77	102

SECTION 4

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

Samples collected and subsequent analysis for this incinerator test are summarized in Table F-3 for each sampling point shown previously in Figure F-1.

During Run 1, there was a wide fluctuation in monitored CO and HC concentrations that coincided in time with waste feed valve cycling. It was also noticed that a flame extended beyond the secondary chamber. Therefore, it was decided that the gas sampling port used for the continuous monitors, Orsat, VOST, and gas bag probes was too close to the secondary chamber. Preceding Run 2, a new sampling port was installed 10.5 ft above the secondary chamber outlet. The four probes were placed in the new port for Runs 2 to 4.

The field crew added 1.5 L each of carbon tetrachloride, trichloroethylene, and chlorobenzene into the waste feed immediately after each drum of waste was dumped into the feed tank. The contents of the tank was then stirred. Only four waste drums were used during the four test runs.

Waste feed subsamples were collected every 15 min from a drain line in the bottom center of the feed tank, which was the only available sampling point. Samples could not be collected from the waste feed line because of the low waste feed flow rate. The actual waste feed line is also located in the bottom of the tank but protrudes 1/2 in. to 1 in. up inside the tank. The method of collecting the feed samples appeared to be less representative of the actual feed material than collection of samples from the feed line. During the tests it was noticed, especially in Runs 3 and 4, that the waste samples contained a large amount of water, even though the incinerator continued to operate normally. This indicates that the waste feed samples probably were not representative. The effect of this situation on test results is discussed in Section 5.

Stack sampling consisted of EPA Method 5 (for particulates and Cl^-) and use of two methods for sampling volatile POHCs; gas bags and a volatile organic sampling train (VOST). Continuous monitors were used for O_2 , CO, and total hydrocarbons. No semivolatile POHC sampling was done because available information on waste feeds indicated that none were present.

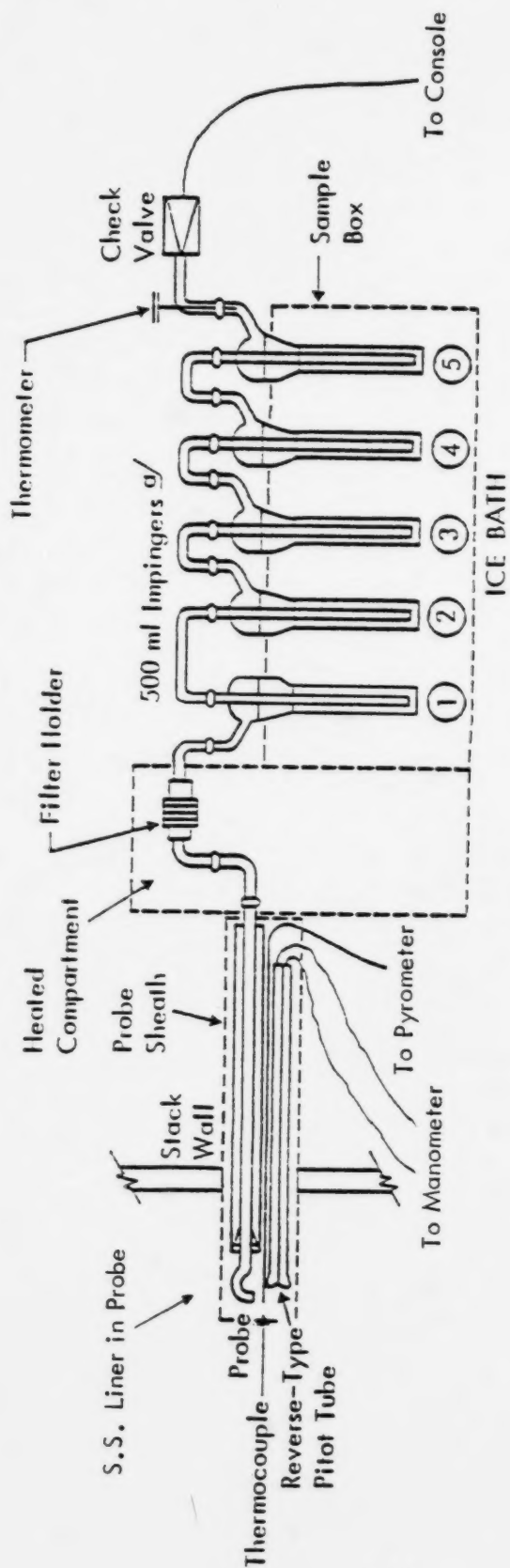
A schematic diagram of the M5 train is shown in Figure F-3 and a diagram of the VOST is shown in Figure F-4.

Gas bag samples were collected in 15-L Tedlar bags, drawing gas into the bags over the test period of about 2 hr.

TABLE F-3. SUMMARY OF SAMPLING AND ANALYSIS

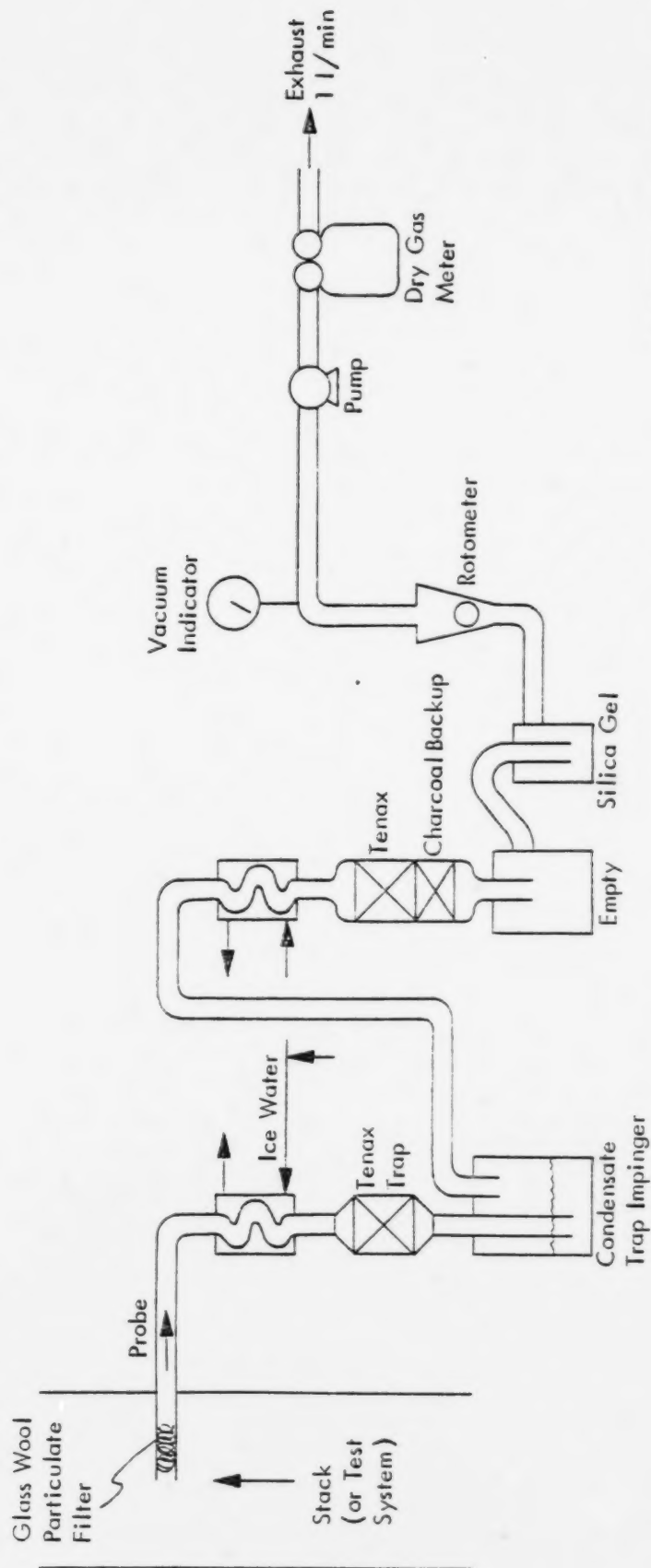
Sampling point	Number of samples per run	Sampling technique	Analysis
1. Waste Feed	1 Composite	Grab samples from feed tank every 15 min	HHV, ash Volatile POHCs Cl, ultimate H ₂ O, viscosity
2. Stack			
M5 Train	1	M5	Particulate weight and Cl ⁻
Volatile Train	1 ^a	VOST Train ^a	Volatile POHCs and PICs
Gas Bags	1	Gas Bags	Volatile POHCs and PICs
CO ₂ and O ₂	1	ORSAT	CO ₂ and O ₂
CO, O ₂ , and HC	Continuous	Continuous Monitors	CO, O ₂ , and HC

^a VOST samples from Run 1 were not analyzed. A VOST sample was not collected during Run 4.



- ① Modified Greenburg-Smith, Reversed, Empty
- ② Greenburg-Smith, 100 ml of Distilled H_2O
- ③ Greenburg-Smith, 100 ml of 0.1 N KOH
- ④ Modified Greenburg-Smith Empty
- ⑤ Modified Greenburg-Smith, SiO_2

Figure F-3. EPA Method 5 particulate sample apparatus.



Both traps were changed
out every 20 minutes over
2 hour period.

Note: Tenax & tenax/charcoal traps were 1.6 cm in diameter
& 10 cm long

Figure F-4. Volatile organic sampling train (VOST).

Gas bags filled with prepure N₂ in the field were also prepared as gas bag blanks.

The VOST was also used, in addition to gas bags, to quantify volatile POHCs. The VOST consists of a pair of Tenax and Tenax/charcoal traps (one each). Gas is drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The pair of traps is then removed and replaced with another pair, until a total of six pairs have been used (i.e., 120 min of sampling time). Blank traps are also utilized, which are traps that are exposed to the sampling location for the same period required to insert sample traps into the VOST apparatus. All traps are then returned to the analytical laboratory for analysis of volatile POHCs and PICs.

POHCs in the waste feed and stack samples were analyzed by GC/MS. The waste feed samples were first homogenized either manually or mechanically depending upon their physical state.

The homogenized feed samples were analyzed for volatile POHCs using a method based on Method A101b in the A. D. Little report entitled "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0-g aliquot of the mixed waste feed sample is dispersed in 40 mL of prepurified tetraglyme. A 10- μ L aliquot of the tetraglyme solution is spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample is then analyzed by the purge-trap-desorb technique as described in Method A101A in the A. D. Little report (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis may be repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with the VOST. A known amount of the gas bag sample, usually 5.0 L, was transferred onto a Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). A needle was inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were analyzed using the same techniques as for the VOST traps described below.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene (BFB) internal standards using the flash vaporization technique in which the spiking solution was vaporized and carried onto the trap with a carrier gas. For spiking, the traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, was turned on through the trap and a syringe needle injected through the GC septum port. One microliter of a 25-ng/ μ L methanolic solution of the internal standards was slowly expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water

column (1 to 5 mL) which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds passed into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Particulate and chloride analyses were conducted according to EPA Method 5 and EPA Method 325.2 from "Methods for Chemical Analysis of Water and Wastes," respectively.

Water content in the waste feeds was determined by the Karl Fischer titration method. Chlorine content was determined by potentiometric titration using a Fisher Titrator Model 41. Heating value was determined by ASTM Methods D240-64. EPA Method 3 was followed when conducting Orsat analysis of the stack effluent. The continuous monitoring instrumentation consisted of the following:

Oxygen - Beckman Model 742 (polarographic sensor)

CO - Beckman Model 215A (nondispersive infrared)

HC - Beckman Model 402 (heated oven flame ionization detector)

More detailed descriptions of all the above sampling and analysis methods are presented in Appendix B.

SECTION 5

DISCUSSION OF RESULTS

INPUT AND OUTPUT CONCENTRATIONS AND RATES FOR POHCs

POHC input and output concentrations and rates were determined for all volatile POHCs listed by EPA which were present in the waste feed at concentrations above 100 $\mu\text{g/g}$. No attempt was made to restrict the number of POHCs based on any selection or ranking method. Since no semivolatile POHCs were predicted to be present in the Zapata waste, only volatile POHCs were analyzed. All concentrations were determined relative to authentic calibration standards.

The concentrations of POHCs in the waste feed are given in Table F-4. Input rates of POHCs are presented in Table F-5, based upon the POHC concentration values in Table F-4 and the waste feed rates presented earlier in Table F-2. Table F-5 presents uncorrected values. Corrections to these values are discussed below.

As mentioned earlier in Section 4, waste feed samples were collected from a drain line at the bottom of the feed tank and not directly from the waste feed line. Observations during the tests indicate that the waste feed samples probably were not representative of the waste in the feed tank, as fed to the incinerator. Table F-6 lists the concentrations of carbon tetrachloride, trichloroethylene, and chlorobenzene in the waste feed based on the rate these compounds were spiked into the waste feed tank. By comparing these values with the results of waste feed analysis in Table F-4 a significant discrepancy is noted, especially in Run 4.

When waste feed subsamples were taken it was apparent, especially in Runs 3 and 4, that there was a large fraction of water in the waste samples. The final composite sample had two distinct layers. At times the subsamples were almost entirely water, but the incinerator continued to operate normally. (The percent water in the waste feed is presented later in this section.)

Based on the above, it was concluded that the waste feed samples were not totally representative of the actual waste feeds. Therefore, Table F-6 presents correction factors indicating the difference between the concentration of compounds as added to the waste, and the actual analysis results. Table F-7 presents the corrected POHC input rates based on the correction factors in Table F-6. Toluene and methylene chloride are also corrected in Table F-7 but an average correction factor based on the three spiked compounds was used. Since the effects of the waste feed sampling method on toluene and methylene chloride analysis results cannot be accurately predicted from the existing data, the input rates for these two compounds are less certain.

TABLE F-4. POHC CONCENTRATIONS IN WASTE FEED
($\mu\text{g/g}$)

	Run 1	Run 2	Run 3	Run 4
Methylene chloride	64	170	< 5	< 5
Carbon tetrachloride	12,000	7,300	6,100	2,800
Trichloroethylene	11,000	7,100	5,200	2,900
Toluene	1,100	3,300	730	4,200
Chlorobenzene	7,800	7,600	7,900	4,000

TABLE F-5. POHC INPUT RATE (UNCORRECTED),^a g/min

	Run 1	Run 2	Run 3	Run 4
Methylene chloride	0.04	0.1	< 0.004	< 0.004
Carbon tetrachloride	7.9	5.5	4.8	2.2
Trichloroethylene	7.3	5.4	4.1	2.2
Toluene	0.7	2.5	0.6	3.2
Chlorobenzene	5.1	5.8	6.2	3.1

^a See corrected POHC input rates in Table F-7.

TABLE F-6. SPIKING CONCENTRATION AND CORRECTION FACTORS^a

	Spiking concentration (µg/g)	Correction factor			
		Run 1	Run 2	Run 3	Run 4
Carbon tetrachloride	13,500	1.1	1.8	2.2	4.8
Trichloroethylene	12,200	1.1	1.7	2.3	4.2
Chlorobenzene	9,400	1.2	1.2	1.2	2.4
Average correction factors		1.1	1.6	1.9	3.8

^a Correction factors = $\frac{\text{Spiking concentration}}{\text{Measured concentration}}$

TABLE F-7. CORRECTED POHC INPUT RATES,^a g/min

	Run 1	Run 2	Run 3	Run 4
Methylene Chloride ^b	0.04	0.16	< 0.008	< 0.02
Carbon tetrachloride	8.7	9.9	11	11
Trichloroethylene	8.0	9.2	9.4	9.2
Toluene ^b	0.8	4.0	1.1	12
Chlorobenzene	6.1	7.0	7.4	7.4

^a Based on correction factors in Table F-6.

^b Based on average correction factor in Table F-6.

Tables F-8 and F-9 present volatile POHC output data (not blank corrected) for both gas bags and VOST samples. (VOST samples were not analyzed for Run 1 and were not collected during Run 4). Table F-8 lists the output concentrations in nanograms per liter (ng/L) and Table F-9 shows the output rates in g/min.

EMISSION OF PICs

Analysis of samples included identification of PICs, i.e., those compounds identified in the stack samples that are listed as POHCs but which were not found in the waste feed samples (i.e., concentrations less than 100 µg/g). The output concentration and rate for these compounds are tabulated in Tables F-10 and F-11.

BLANK CORRECTION DATA FOR POHCs AND PICs

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table F-12. The sample volumes for gas bags as well as VOST are presented in Table F-13.

DREs FOR POHCs

Destruction and removal efficiencies (DREs) based on the above data are presented in Table F-14. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$\text{DRE} = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

PARTICULATE EMISSIONS

The particulate emission data are presented in Table F-15. Associated Method 5 test data are given in Table F-16. The particulate emission data for all four runs show that the particulate concentration was less than the allowable concentration of 180 mg/dscm (corrected to 7% O₂) specified in EPA's regulation.

CONTINUOUS MONITORING DATA

Continuous monitors were used to determine concentrations of O₂, CO, and HC in the stack effluent throughout the test period. The results are summarized in Table F-17. The results for Run 1 were affected by the lower position of the sampling port for that run, as discussed previously. The CO and HC concentrations for Runs 2, 3, and 4 were significantly lower.

HCl EMISSION RATES

Table F-18 lists the Cl⁻ emission rates from the stack. The data show that the output rate of Cl⁻ was less than 1.8 kg/hr for each of the four runs in accordance with current EPA regulations. Part of the Cl⁻ in the stack was due to the compounds spiked into the waste.

TABLE F-8. STACK OUTPUT CONCENTRATIONS OF VOLATILE POHCs (NOT BLANK CORRECTED), ng/L

	Gas bags				VOST ^b							
	Run 1 ^a	Run 2		Run 3	Run 4	Run 2		Run 3		6th Pair	3rd Pair	6th Pair
		Run 1	Run 2			1st Pair	3rd Pair	1st Pair	3rd Pair			
Methylene chloride	76	15		5.4	4.8	12		c		2.6	7.0	2.5
Carbon tetrachloride	114	19		308	14	5.7		2.0		3.6	6.6	5.2
Trichloroethylene	80	56		84	22	20		3.2		12	4.4	4.3
Toluene	18	28		18	24	45		1.1		4.6	2.5	3.1
Chlorobenzene	13	12		30	6	41		1.5		1.9	22	3.1

^a The volatile POHC sampling point was relocated higher up the stack for Runs 2, 3, and 4.

^b There is no VOST data for Runs 1 and 4.

^c Data missed during GC/MS acquisition.

TABLE F-9. STACK OUTPUT RATES FOR VOLATILE POHCs (NOT BLANK CORRECTED), g/min

	Gas bags				VOST ^b			
	Run 1 ^a	Run 2	Run 3	Run 4	1st Pair	6th Pair	3rd Pair	6th Pair
Ethylene chloride	0.00016	0.00012	0.00011	0.00011	0.00026	0.000075	0.000058	0.000054
Carbon tetrachloride	0.0024	0.00041	0.00068	0.00031	0.00013	0.000045	0.000091	0.00011
Trichloroethylene	0.0017	0.0012	0.0019	0.00048	0.00044	0.000070	0.000099	0.000098
Toluene	0.00034	0.00060	0.00040	0.00053	0.0010	0.000025	0.00010	0.000055
Chlorobenzene	0.00028	0.00026	0.00066	0.00013	0.00091	0.000033	0.000042	0.000068

^a The volatile POHC sampling point was relocated higher up the stack for Runs 2, 3, and 4.

^b There is no VOST data for Runs 1 and 4.

^c Data missed during GC/MS acquisition.

	Gas bags						VUST							
	Run 1		Run 2		Run 3		Run 4		Run 2		Run 3			
	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	Run 1	Run 2	1st Pair	3rd Pair	6th Pair	1st Pair	3rd Pair	6th Pair
Chloroform	1.7	1.6	1.6	1.6	1.4	1.4	1.5	1.1	5.0	3.2	1.2	4.0		
1,1,1-Trichloroethane	1.8	2.4	1.2	1.2	1.2	1.2	0.41	0.31	0.92	0.71	1.2	0.81		
Benzene	36	58	7.4	30	110	110	2.3	5.9	8.3	18	10			
Tetrachloroethylene	2.0	1.0	1.0	1.0	1.0	1.0	0.87	0.47	0.56	0.96	0.66	1.1		

	Gas bags								VOST					
	Run 1		Run 2		Run 3		Run 4		Run 2		Run 3		Run 4	
	1st Pair	2nd Pair	1st Pair	2nd Pair	1st Pair	2nd Pair	1st Pair	2nd Pair	1st Pair	2nd Pair	1st Pair	2nd Pair	1st Pair	2nd Pair
Chloroform	0.000036	0.000035	0.000035	0.000035	0.000035	0.000035	0.000031	0.000031	0.000034	0.000024	0.000011	0.000071	0.000027	0.000089
1, 1, 1-Trichloroethane	0.000038	0.000052	0.000027	0.000026	0.000026	0.000026	0.000026	0.000026	0.0000090	0.0000069	0.000020	0.000016	0.000027	0.000018
Benzene	0.000072	0.0013	0.00016	0.00066	0.00066	0.00066	0.0024	0.00052	0.00013	0.00018	0.00040	0.00022	0.00022	0.00022
Tetrachloroethylene	0.000042	0.000022	0.000022	0.000022	0.000022	0.000022	0.000019	0.000010	0.000012	0.000021	0.000015	0.000024	0.000024	0.000024

TABLE F-12. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>				
Methylene chloride	20	3	35	46
Carbon tetrachloride	110	90	< 1	0
Trichloroethylene	300	70	4.5	3.7
Toluene	87	19	45	47
Chlorobenzene	27	14	9	14
<u>PICs</u>				
Chloroform	4	1	60	70
1,1,1-Trichloroethane	5	1	0.5	1
Benzene	20	8	74	62
Tetrachloroethylene	3	1	4.8	4.8

TABLE F-13. VOLATILE SAMPLE VOLUMES (DRY
STANDARD LITERS)

Gas bags		VOST	
Run No.	Volume (L)	Run No./pair No.	Volume (L)
1	5.0	2/1	19.6
2	5.0	2/3	19.2
3	5.0	2/6	19.6
4	5.0	3/1	19.8
		3/3	19.5
		3/6	19.8

TABLE F-14. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs) FOR THE ZAPATA INCINERATOR
(BLANK CORRECTED)

Volatile compounds	Gas bags					Fast VOST	
	Run 1	Run 2	Run 3	Run 4	Run 2	Run 3	
Methylene chloride	a	99.84	a	a	> 99.906	a	
Carbon tetrachloride	99.978	> 99.9957	99.943	> 99.9972	99.99911	99.9990	
Trichloroethylene	> 99.979	> 99.987	> 99.976	> 99.9946	99.9979	99.9985	
Toluene	> 99.952	> 99.985	> 99.965	> 99.9956	> 99.9914	> 99.9932	
Chlorobenzene	> 99.9956	> 99.9963	99.9927	> 99.9983	> 99.9953	> 99.9974	

a Waste feed concentration < 100 µg/g in this run.

TABLE F-15. PARTICULATE EMISSION DATA

	Particulate concentration			
	Run 1	Run 2	Run 3	Run 4
Measured				
mg/dscm	37.8	31.8	57.3	27.1
(gr/dscf)	(0.0165)	(0.0139)	(0.0250)	(0.0118)
Corrected ^a				
mg/dscm	69.1	50.2	81.9	38.5
(gr/dscf)	(0.0301)	(0.0219)	(0.0357)	(0.0168)
Emission rate	Particulate emissions			
	Run 1	Run 2	Run 3	Run 4
kg/hr	0.05	0.04	0.08	0.04
(lb/hr)	(0.11)	(0.09)	(0.17)	(0.08)

^a Measured emissions were corrected to 7% O₂ as follows:

$$C_{\text{corr}} = \frac{(14\%)}{(21\% - \%O_2)} (C_{\text{measured}})$$

TABLE F-16. METHOD 5 TEST DATA

	Run 1	Run 2	Run 3	Run 4
Vol. of gas sampled, Nm ³ (dscf)	1.840 (64.981)	1.962 (69.291)	2.018 (71.258)	1.969 (69.532)
Sampling time (min)	144	144	144	144
Percent isokinetic	96.9	101.2	102.1	100.2
Moisture content (%)	8.7	9.7	11.1	10.5
Percent O ₂ (dry)	13.3	12.1	11.2	11.1
Percent CO ₂ (dry)	5.8	6.6	6.5	6.7
Stack temperature, °C (°F)	663 (1,225)	611 (1,132)	646 (1,195)	679 (1,254)
Stack flow rate, act. m ³ /min (act. ft ³ /min)	75 (2,642)	73 (2,565)	78 (2,767)	80 (2,825)
Stack flow rate, Nm ³ /min (dscf/min)	21 (749)	22 (765)	22 (780)	22 (775)

TABLE F-17. CONTINUOUS MONITORING DATA (STACK)^a

	Run 1 ^b 9/28/82	Run 2 9/29/82	Run 3 9/29/82	Run 4 9/30/82
Oxygen (%)				
Range	7.2-9.0	10.1-13.2	10.0-14.4	10.8-13.1
Average	8.2	12.0	11.8	11.9
Carbon monoxide (ppm)				
Range	739-1,570	< 1-394	3.6-8.8	2.8-18.3
Average	1,275	22.2	4.7	8.8
Total hydro- carbons (ppm) ^c				
Range	32-215	< 1-40.9	< 1	< 1-2.9
Average	71	1.9	< 1	< 1

^a Concentrations on dry gas basis.

^b The sampling point was relocated higher up in the stack for Runs 2, 3, and 4.

^c Total HC reported as propane.

TABLE F-18. STACK Cl⁻ EMISSION RATES

	kg/hr of Cl ⁻	lb/hr of Cl ⁻
Run 1	1.01	2.23
Run 2	0.63	1.39
Run 3	1.25	2.75
Run 4	1.50	3.30

ANALYSIS OF WASTE FEED

Table F-19 summarizes data on percent chlorine, percent H₂O, viscosity, and heating value for the waste feed. The water content increased significantly in the waste feed in Runs 3 and 4. In response, the heating value and chlorine content decreased in Runs 3 and 4. As discussed earlier, it is believed that the waste feed samples were not representative of the waste fed to the incinerator, especially in Runs 3 and 4.

The results of an ultimate analysis for the waste feed in Run 1 are presented in Table F-20.

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (1) external auditing; (2) sampling QA activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. A comprehensive performance audit (quantitative evaluation) and limited systems audit (qualitative evaluation) were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on:

- GC/MS analysis of volatile organics; and
- Technicon Autoanalyzer analysis of chlorides.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics. Analyses of chloride audit samples were also determined to be adequate and acceptable.

Systems audits demonstrated that laboratory analyses were done according to protocol.

Sampling QA Activities

In general, sampling QA activities are scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were

TABLE F-19. GENERAL ANALYSIS OF WASTE FEED

	Percent Cl	Percent water	Percent ash	Heating value kJ/kg (Btu/lb)
Run 1	2.69	0.68	0.018	37,544 (16,155)
Run 2 ^a	1.62	0.63	0.013	37,891 (16,304)
Run 3	0.29	1.34	0.0098	22,796 (9,809)
Run 4	0.87	0.74	0.14	15,227 (6,552)

^a The viscosity of the waste in Run 2 was measured as 106.8 SUS at 100°F.

TABLE F-20. ULTIMATE ANALYSIS OF WASTE FEED

	Percent Cl	Percent C	Percent H	Percent N	Percent S	Percent ash	Percent O (by difference)
Run 1	2.69	79.75	9.96	0.036	0.74	0.018	6.81

performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures included spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure used was monitoring the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX G

SUMMARY OF RESULTS FROM TESTING AT
THE AMERICAN CYANAMID INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration regulations. Emissions data for the RIA are being provided by a sampling and analysis program. EPA selected the hazardous waste incinerator at American Cyanamid Company, as one facility for emission measurements.

Five test runs were conducted at this site during the period October 26 to 30, 1982. Samples of waste feeds and gaseous effluents were collected and analyzed. The following sections of this appendix present a summary of results, a brief process description, a synopsis of the test plan and sampling and analysis methods, and a discussion of the test results.

SECTION 2

SUMMARY OF RESULTS

A major part of the effort on this test program was determination of principal organic hazardous constituents (POHCs) in the waste feeds, and quantification of those compounds and products of incomplete combustion (PICs) in the stack outlet gas. The POHCs and PICs were divided into two groups: volatiles and semivolatiles. No volatile POHCs were detected at a concentration above 100 µg/g in the waste feeds. However, the following semivolatile POHCs were detected in the two different production waste streams, which are burned separately:

Aniline waste

Aniline
Phenylenediamine
Diphenylamine

Mononitrobenzene waste

Aniline
Mononitrobenzene
M-Dinitrobenzene

Destruction and removal efficiencies, along with other data involving these compounds, are presented in Section 5. Compounds identified as PICs in the effluent gas were:

Volatiles

Chloroform
Benzene
Toluene
1,1,1-Trichloroethane
Carbon tetrachloride
Trichloroethylene
Tetrachloroethylene
Chlorobenzene

Semivolatiles

Naphthalene
O-Nitrophenol

This test program also included determination of particulate, chloride, and metal emissions. Results of the particulate measurements showed that the outlet particulate concentration exceeded the EPA regulation for hazardous waste incinerators of 180 mg/dscm (0.08 gr/dscf)* only during test Run 5. Runs 1 through 4 were below that concentration level.

Chloride emissions averaged 0.003 kg/hr (0.007 lb/hr) which is less than the EPA emission limit of 1.8 kg/hr (4.0 lb/hr).

* Corrected to 7% O₂.

Iron was found to be the major metal present in the particulate emissions (22%). Chromium and nickel were also found to be present in the waste feed and outlet particulate as follows:

	Conc. in waste feed ($\mu\text{g/g}$)	Conc. in particulate ($\mu\text{g/g}$)
Chromium	250	47,000
Nickel	170	49,000

SECTION 3

PROCESS DESCRIPTION AND PROCESS DATA

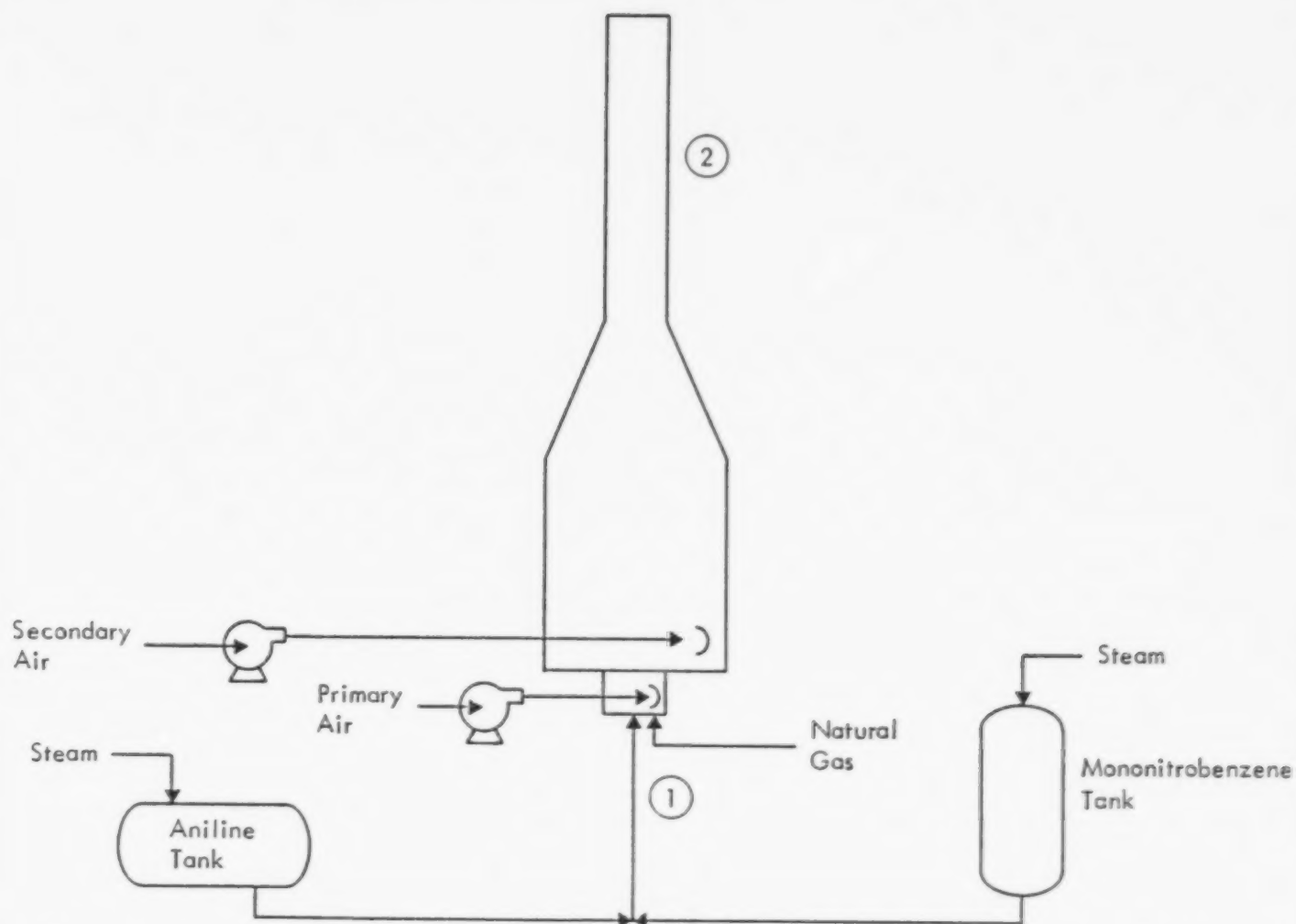
The incinerator is an industrial facility located in West Virginia. It has a small chamber to mix the primary air with waste followed by the combustion chamber. The system does not have any emission control devices (see Figure G-1). Waste and primary air enter the small mixing chamber and pass into the combustion chamber where secondary air is added. Exhaust gases from the combustion chamber enter the stack. A drawing of the combustion chamber, including the location of the thermocouple (T/C), is presented in Figure G-2.

Since the burn period for either unit was so short (1 to 2 hr), it was necessary to start each test as soon as the unit had been started up and was at normal operating conditions (approximately 30 min). The unit was always started on natural gas and run for about 15 min. Waste feed flow was then initiated, and normally increased as the natural gas flow was reduced and finally shut off entirely. Final adjustments were then made in the waste feed flow, after which testing was started.

The facility burns two different waste streams--an aniline waste and a mononitrobenzene waste. Aniline waste collects in a 100-gal. pressurized tank and is fed to the incinerator by steam pressure once a day. Each day's supply of aniline waste is burned during a 1-1/2- to 2-hr period. Mononitrobenzene waste is handled similarly except that the tank has a 50-gal. capacity and the waste is burned during a one hour period, once every 7 to 10 days. The wastes were burned separately in the test program, which was the plants normal practice.

Process data obtained during the tests are summarized in Table G-1, along with information needed for dispersion modeling. These data show that the average temperature in the lower section of the stack ranged from 629 to 779°C and the calculated residence time in the secondary chamber was 0.17 to 0.24 sec. Average heat input to the incinerator was 4.7×10^6 kJ/hr (4.5×10^6 Btu/hr).

The incinerator was operated normally during all five test runs. However, cyclonic flow was noted during a preliminary stack velocity traverse which necessitated the installation of a flow straightener in the lower part of the stack prior to Run 2. Thus, only Run 1 was performed without the flow straightener in place.



Note: Natural Gas is burned only during startup. Aniline and mononitrobenzene waste feeds are always burned separately.

Figure G-1. Diagram of process and sampling locations.

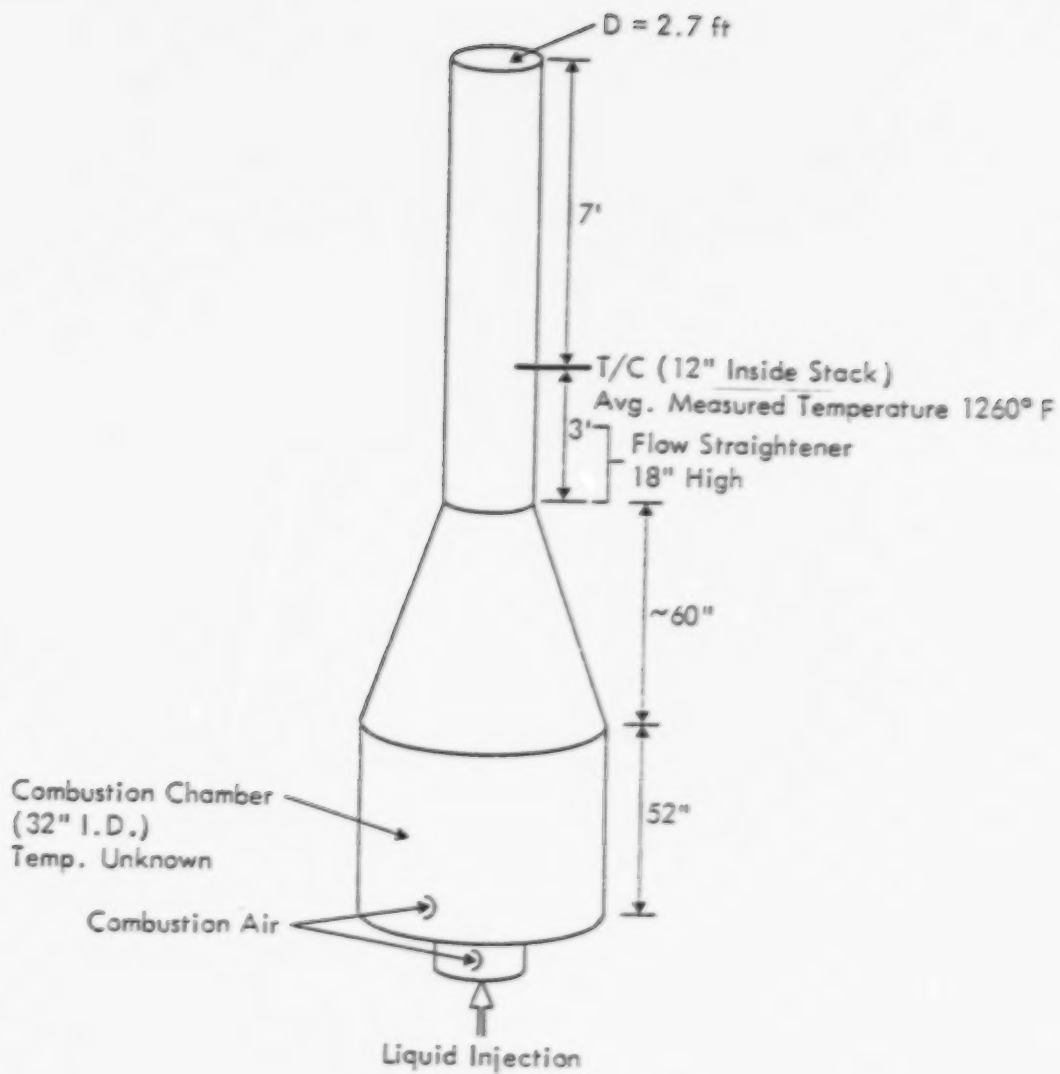


Figure G-2. Combustion chamber diagram.

TABLE G-1. PROCESS DATA^a

	Aniline waste				Mononitro- benzene waste
	Run 1, ^b 10/26/82	Run 2, 10/28/82	Run 3, 10/29/82	Run 5, 10/30/82	Run 4, ^b 10/29/82
Calculated resi- dence time, sec	0.17	0.21	0.24	0.21	0.23
Stack temperature 3 ft from bottom of stack, °C (°F)	779 (1434)	671 (1240)	629 (1164)	648 (1198)	679 (1254)
Feed pressure to nozzle, kPa (psig)	190 (27)	96 (14)	96 (14)	210 (30)	210 (30)
Atomizing steam pressure, kPa (psig)	210 (30)	120 (18)	130 (19)	280 (40)	280 (40)
Feed tank pressure, kPa (psig)	410 (60)	430 (63)	470 (69)	360 (53)	576 (84)
Mixing chamber pressure, kPa (in H ₂ O)	4.0 (16)	3.0 (12)	2.7 (11)	3.0 (12)	3.0 (12)
Energy input, 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	4.9 (4.7)	5.1 (4.8)	4.5 (4.2)	4.5 (4.3)	4.7 (4.5)
Stack exit velocity, m/min (ft/min)	450 (1470)	330 (1070)	330 (1070)	350 (1140)	370 (1210)
Stack temperature at sampling location, °C (°F)	699 (1290)	543 (1010)	576 (1070)	605 (1120)	640 (1180)
Stack height, m (ft)	7.6 (25)	7.6 (25)	7.6 (25)	7.6 (25)	7.6 (25)

^a Average of readings taken during each run.

^b Run 4 was conducted when feeding mononitrobenzene waste. All other runs were on aniline waste. Run 1 was conducted prior to installation of a flow straightener in lower part of the stack.

^c Residence time calculated from volume of secondary chamber, stack flow rate, and temperature measured at thermocouple in lower part of stack.

SECTION 4

TEST PLAN AND ANALYSIS METHODS

Samples collected and subsequent analysis for this incinerator test are summarized in Table G-2 for each sampling point shown previously in Figure G-1. As mentioned in Section 3, a flow straightener was installed in the lower part of the stack prior to Run 2 to prevent cyclonic flow for Runs 2 through 5.

Unlike previous tests in this series of incinerator tests, chemicals were not spiked into the waste feed for testing purposes. The normal plant waste feeds were used during the test periods.

During each run, subsamples (~100mL) of the waste feed to the incinerator were taken every 15 min and composited to obtain a representative sample of the waste burned. Samples were collected from a tap in the waste feed line. Waste feedrates for the aniline wastes were determined by using a measuring rod to determine the level in the feed tank before and after each run. Waste feedrates for the mononitrobenzene waste were determined by reading the tank level, via a sight glass level gage in the tank.

Stack sampling consisted of Modified Method 5 for particulates, Cl^- , metals, and semivolatile POHCs and two methods for sampling volatile POHCs; gas bags and a volatile organic sampling train (VOST). Two Method 5 trains were used simultaneously (one in each port) during each test because the incinerator operating period was short (approximately 1 hr). Continuous monitors were used to measure O_2 , CO_2 , CO, and total hydrocarbons.

A schematic diagram of the MM5 train is shown in Figure G-3 and a diagram of the VOST is shown in Figure G-4.

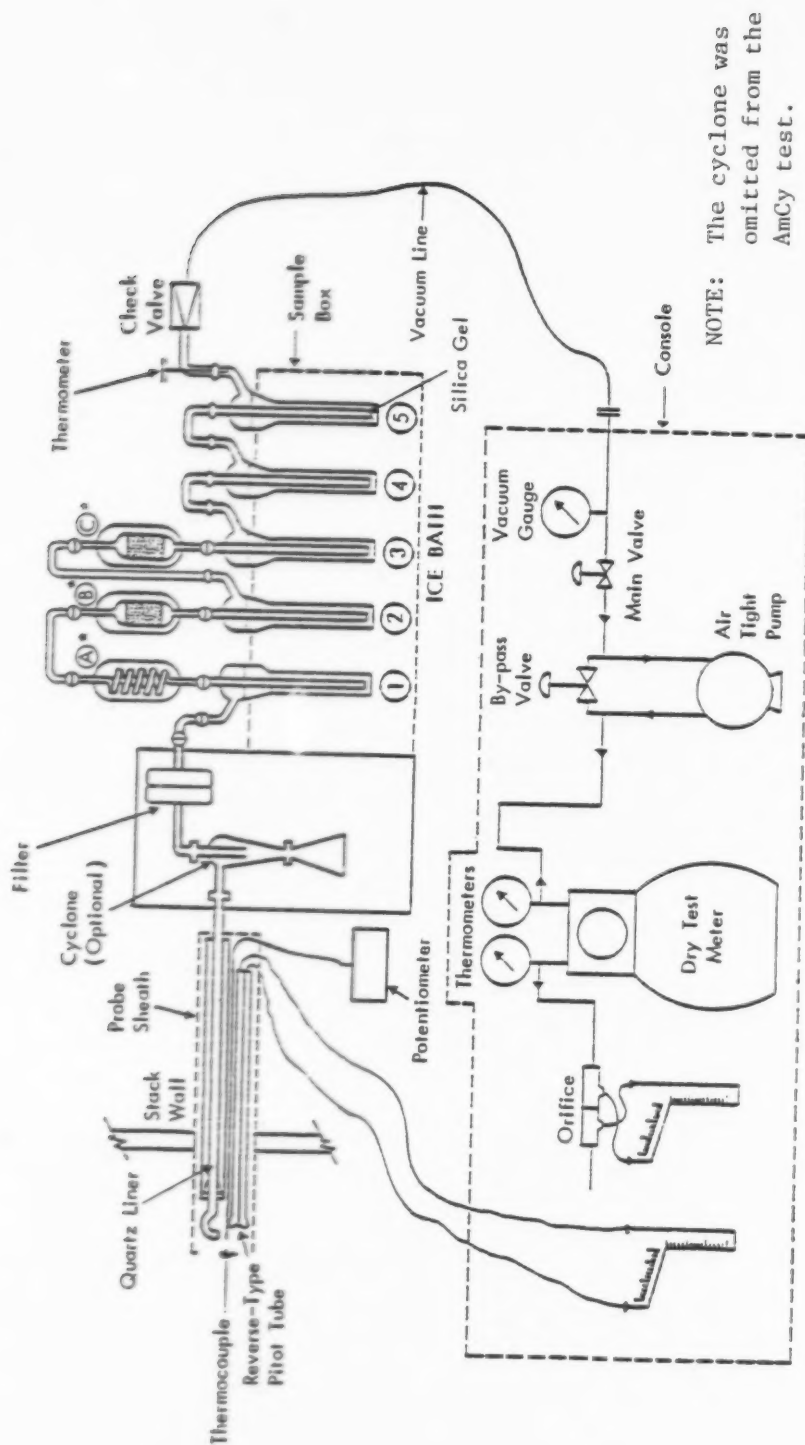
Gas bag samples were collected in 15-L Tedlar bags, drawing gas into the bags over the test period of about 1 hr. Gas bags filled with prepure N_2 in the field were also prepared as gas bag blanks.

The VOST was also used, in addition to gas bags, to quantitate volatile POHCs and PICs. The VOST consists of a pair of Tenax and Tenax/charcoal traps (one each). Gas is drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The pair of traps are then removed and replaced with another pair, and so on until the test was completed (which was only about 1 hr, since the amount of waste was limited). Blank traps which are exposed to the sampling location for the same period required to insert sample traps into the VOST apparatus are also utilized. All traps are then returned to the analytical laboratory for analysis of volatile POHCs and PICs.

TABLE G-2. SUMMARY OF SAMPLING AND ANALYSIS

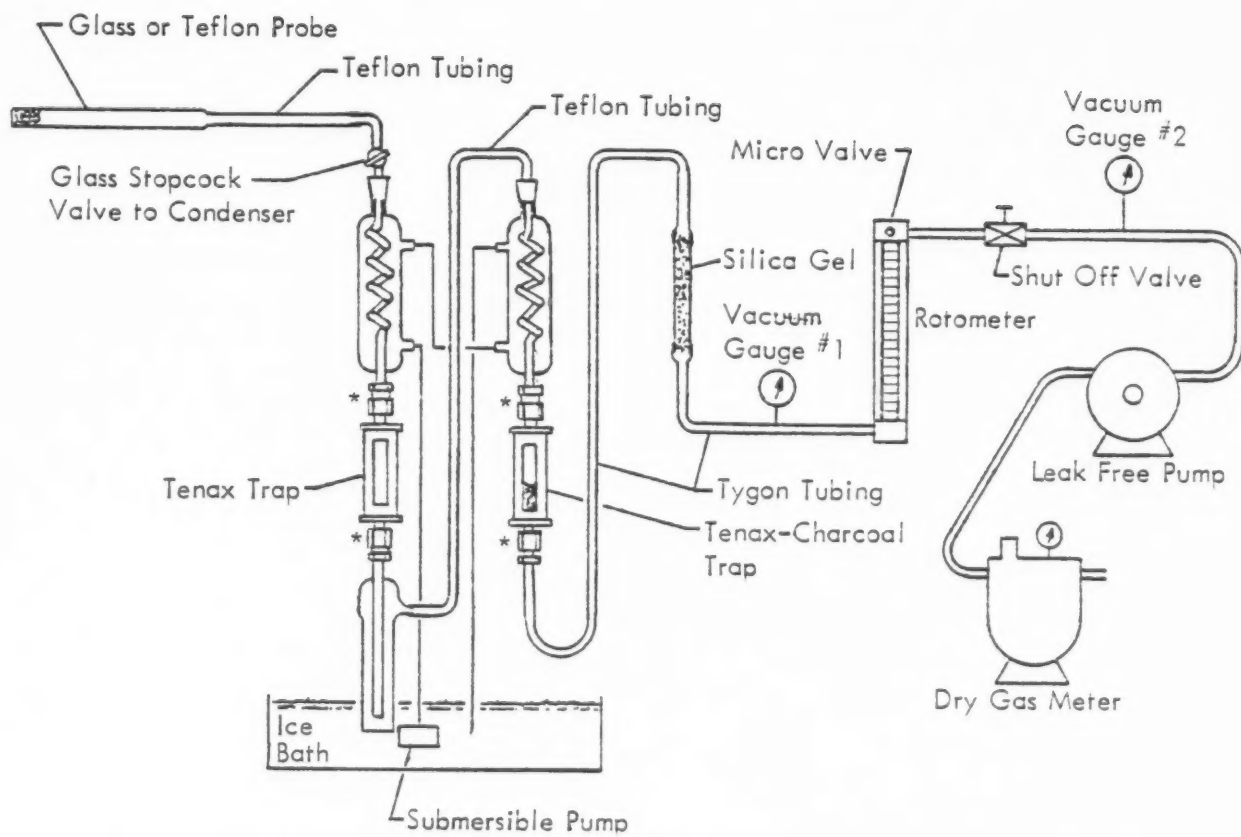
Sampling point	No. of samples per run	Sampling technique	Analysis
1. <u>Waste feed</u>			
Organic waste	1 Composite	Grab samples every 15 min	HHV POHCs Cl ⁻ H ₂ O Metals ^a Ash
2. <u>Stack</u>			
MM5 train	2	2 MM5 trains (each train traversed one stack diameter)	Particulate weight Semivolatile POHCs and PICs Cl ⁻ Metals ^a
Volatile train	1	VOST train	Volatile POHCs and PICs
	1	Gas bag	
CO ₂ and O ₂	1	Orsat	CO ₂ and O ₂
CO, CO ₂ , O ₂ , and HC	Cont.	Continuous monitors	CO, CO ₂ , O ₂ , and HC

^a Only samples from Run 3 were analyzed for metals.



- ① Modified Greenburg-Smith, Reversed, Empty
 ② Greenburg-Smith, 50 ml of Double Distilled in Glass H₂O
 ③ Greenburg-Smith, 100 ml of 0.1 N KOH
 ④ Modified Greenburg-Smith Empty
 ⑤ Modified Greenburg-Smith, SiO₂
- (A) Condenser
 (B) XAD Resin Cartridge #1
 (C) XAD Resin Cartridge #2
 * Ice Water Jacket

Figure G-3. Modified Method 5 sampling train (NM5).



* Special O-Ringed Stainless Steel Fittings

Figure G-4. Volatile organic sampling train (VOST).

POHCs in the waste feed and stack samples were analyzed by GC/MS. The waste feed samples were first homogenized either manually or mechanically depending on their physical state.

The homogenized feed samples were analyzed for volatile POHCs using a method based on Method A101b in the A. D. Little report entitled "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0-g aliquot of the mixed waste feed sample is dispersed in 40 mL of prepurified tetraglyme. A 10- μ L aliquot of the tetraglyme solution is spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample is then analyzed by the purge-trap-desorb technique as described in Method A101a in the A. D. Little report (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis may be repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs are analyzed in the waste feed samples according to Method P023 of the A. D. Little report feed sample is spiked with semivolatile surrogate compounds, such as decafluorobuphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 μ g/g depending on the expected sample complexity. The sample is allowed to equilibrate with the spiked compounds and then is extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appears on the surface of the methylene chloride, the pH is checked and it is neutralized. The extract is then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

An aliquot of the extract was then screened by gas chromatography with a flame ionization detector. If necessary, the extract was further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column (FSCC). A 1.0-mL aliquot of the extract was then spiked with 20 μ g of D₁₀-anthracene internal standard and analyzed by FSCC GC/MS.

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with VOST. A known amount of the gas bag sample, usually 5.0 L, was transferred onto a Tenax/charcoal trap at the analytical laboratory (the same type of trap used as the backup trap in the VOST). This transfer was done by inserting a needle into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were then analyzed using the same techniques as for the VOST traps described below.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene (BFB) internal standards using the flash vaporization technique in which the spiking solution was vaporized and carried onto the trap with a carrier gas. In order to do this, the glass traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, was turned on through the trap and a syringe needle injected through the GC septum port. One μ L of a 25-ng/ μ L methanolic solution of the internal standards was slowly

expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was only about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL) which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds pass into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs involves several components of the MM5 train. Each of the components of the MM5 train were spiked with a separate surrogate standard according to the following scheme.

Front XAD-2 resin trap: 20 µg of ¹³C-tetrachlorobiphenyl
Back XAD-2 resin trap: 20 µg of decafluorobiphenyl
Particulate filter plus probe rinse residue: 20 µg of D₁₂-chrysene
Condensate water: 20 µg of D₈-naphthalene

The spiked components were normally extracted separately, and then the extracts were combined, dried with anhydrous sodium sulfate, concentrated to 1.0 ml using Kuderna-Danish evaporation according to Method P031 in the A. D. Little report and using supplementary gentle nitrogen blowdown, and analyzed by FSCC GC/MS. The samples were extracted according to the following procedures:

XAD-2 resin traps: The XAD-2 resin traps were Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Particulate filters plus probe rinse residues: The residue from the acetone rinse of the probe was added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Condensate water: The condensate water was extracted with three 60-ml aliquots of methylene chloride as described in Method P021a in the A. D. Little report (equivalent to EPA Method 625).

Metals analysis was by acid digestion of samples and analysis by inductively coupled argon plasma emission spectrophotometry and/or atomic absorption spectrophotometry. Particulate and chloride analyses were conducted according to EPA Method 5 and EPA Method 325.2 from "Methods for Chemical Analysis of Water and Wastes," respectively. Water content in the waste feeds was determined by the Karl Fischer titration method. Chlorine in the waste feed was determined by combusting the sample in a Schoniger blank followed by potentiometric titration using a Fisher Titralizer Model 41. Heating value was determined by ASTM Method D240-64. EPA Method 3 was followed

when conducting Orsat analysis of the stack effluent. Continuous monitoring instrumentation consisted of the following.

- Oxygen - Beckman Model 742 (polarographic sensor)
- CO - Beckman Model 215A (nondispersive infrared)
- HC - Beckman Model 402 (heated oven flame ionization detector)
- CO₂ - Horiba Model PIR-2000S (nondispersive infrared)

More detailed descriptions of all the above sampling and analysis methods are presented in Appendix B.

SECTION 5

DISCUSSION OF RESULTS

INPUT AND OUTPUT CONCENTRATIONS AND RATES FOR POHCs

POHC input and output concentrations and rates were determined for all POHCs listed by EPA which were found to be present in the waste feeds at concentrations above 100 $\mu\text{g/g}$. No attempt was made to restrict the number of POHCs based on any selection or ranking method. However, no volatile POHCs were detected in the waste feed in any of the five runs. In all tables, values marked < indicate detection levels.

The waste feed rates are given in Table G-3 and the concentrations of semivolatile POHCs in the waste feeds are given in Table G-4. Input rates of these POHCs are presented in Table G-5, based on the waste feed rates and POHC concentration values.

Tables G-6 and G-7 present semivolatile POHC output data (not blank corrected). The data for each POHC in each test run are presented for the front half and back half separately. Front half refers to the probe rinse and filters in the MM5 trains and thus addresses the POHCs in the particulate. Back half refers to XAD resins and condensate in the MM5 trains and thus, includes primarily the POHCs present in a gaseous state in the sampling train. Table G-6 lists the output concentrations in ng/L and Table G-7 shows the output rates in g/min .

TABLE G-3. WASTE FEED RATES, kg/min (lb/min)

	Run 1	Run 2	Run 3	Run 5	Run 4
Aniline waste	2.45 (5.39)	2.52 (5.54)	2.22 (4.88)	2.25 (4.95)	0
Mononitrobenzene waste	0	0	0	0	3.17 (6.97)

TABLE G-4. CONCENTRATION OF POHCs IN WASTE FEED ($\mu\text{g/g}$)

	Aniline				Mono-nitrobenzene Run 4
	Run 1	Run 2	Run 3	Run 5	
<u>Volatile POHCs</u>	----- All < 100 $\mu\text{g/g}$ -----				
<u>Semivolatile POHCs</u>					
Aniline	a	550,000	600,000	530,000	8,000
Phenylenediamine	a	2,300	5,300	4,600	< 100
Diphenylamine	a	6,200	5,800	5,400	< 100
Mononitrobenzene	a	< 100	< 100	< 100	640,000
m-Dinitrobenzene	a	< 100	< 100	< 100	3,100

^a Samples from Run 1 were not analyzed for semivolatile POHCs because this run occurred prior to installation of the flow straightener in the stack.

TABLE G-5. CALCULATED INPUT RATES FOR POHCs IN WASTE FEEDS (g/min)

	Aniline			Mono-nitrobenzene Run 4
	Run 2	Run 3	Run 5	
<u>Volatile POHCs</u>	----- None detected -----			
<u>Semivolatile POHCs</u>				
Aniline	1,390	1,330	1,190	30
Phenylenediamine	5.8	12	10	a
Diphenylamine	16	13	12	a
Mononitrobenzene	a	a	a	2,030
m-Dinitrobenzene	a	a	a	10

^a Not detected in waste feed (i.e., < 100 $\mu\text{g/g}$).

TABLE G-6. SEMIVOLATILE POHC CONCENTRATIONS IN STACK
(NOT BLANK CORRECTED), ng/L

	Aniline			Mono- nitro- benzene
	Run 2	Run 3	Run 5	Run 4
<u>Aniline</u>				
Front half	< 0.6	< 0.7	< 0.3	< 0.5
Back half	< 1.5	< 2	< 2	< 3
<u>Phenylenediamine</u>				
Front half	< 0.6	< 0.7	< 0.3	a
Back half	< 1.5	< 2	< 2	a
<u>Diphenylamine</u>				
Front half	< 0.6	< 0.7	< 0.3	a
Back half	< 1.5	< 2	< 2	a
<u>Mononitrobenzene</u>				
Front half	a	a	a	< 0.6
Back half	a	a	a	32
<u>m-Dinitrobenzene</u>				
Front half	a	a	a	< 4
Back half	a	a	a	< 20

Note: Front half refers to probe rinse and filters in MM5 trains.
Back half refers to XAD resins and condensate in MM5 trains.

^a These compounds were not found in waste feed or in stack emissions.

TABLE G-7. SEMIVOLATILE POHC OUTPUT RATES (NOT BLANK CORRECTED),
g/min

	Run 2	Aniline Run 3	Run 5	Mono- nitro- benzene Run 4
<u>Aniline</u>				
Front half	< 0.00003	< 0.00004	< 0.00002	< 0.00003
Back half	< 0.000086	< 0.0001	< 0.0001	< 0.0001
Total	< 0.0001	< 0.0001	< 0.0001	< 0.0001
<u>Phenylenediamine</u>				
Front half	< 0.00003	< 0.00004	< 0.00002	a
Back half	< 0.000086	< 0.0001	< 0.0001	a
Total	< 0.0001	< 0.0001	< 0.0001	
<u>Diphenylamine</u>				
Front half	< 0.00003	< 0.00004	< 0.00002	a
Back half	< 0.000086	< 0.0001	< 0.0001	a
Total	< 0.0001	< 0.0001	< 0.0001	
<u>Mononitrobenzene</u>				
Front half	a	a	a	< 0.00003
Back half	a	a	a	0.0018
Total				0.0018
<u>m-Dinitrobenzene</u>				
Front half	a	a	a	< 0.0002
Back half	a	a	a	< 0.001
Total				< 0.001

Note: Front half refers to probe rinse and filters in MMS trains.
Back half refers to XAD resins and condensate in MMS trains.

^a These compounds were not found in waste feed or in stack emissions.

EMISSION OF PICs

Analysis of samples included identification of PICs, i.e., those compounds identified in the stack samples that are listed as POHCs but which were not found in any of the waste feed samples at concentrations above 100 µg/g. Tables G-8 and G-9 present the output concentration and rates for both volatile and semivolatile PICs. Tables G-10 and G-11 present corresponding output data for volatile PICs.

BLANK VALUES FOR POHCs AND PICs

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table G-12. The sample volumes for gas bags as well as VOST are presented in Table G-13. Blank correction data for semivolatile POHCs and PICs are presented in Table G-14. Sample volumes for the MM5 train can be found in Table G-17.

DREs FOR POHCs

Destruction and removal efficiencies (DREs) based on the above data are presented in Table G-15. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$DRE = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

PARTICULATE EMISSIONS

The particulate emission data are presented in Table G-16. Associated Method 5 test data are given in Table G-17. The particulate emission data show that the particulate concentration was below the allowable concentration of 180 mg/dscm (corrected to 7% O₂) specified in EPA's regulation, in all runs except Run 5.

CONTINUOUS MONITORING DATA

Continuous monitors were used to determine concentrations of O₂, CO₂, CO, and total hydrocarbon (HC) in the stack effluent throughout each test period (an electrical problem shut down all monitors except HC during Run 3). The results are summarized in Table G-18. The concentrations of CO and HC were low throughout the test period.

HCl EMISSION RATES

Table G-19 lists the Cl⁻ emission rates from the stack. The data show that the output rate of Cl⁻ in each run was quite low reflecting the fact that the waste feeds were low in chlorine content.

TABLE G-8. STACK OUTPUT SEMIVOLATILE PIC CONCENTRATIONS
(NOT BLANK CORRECTED), ng/L

	Run 1	Run 2	Run 3	Run 5	Run 4
Naphthalene					
Front half	a	< 0.2	< 0.2	< 0.1	< 0.2
Back half	a	210	25	72	160
O-Nitrophenol					
Front half	a	< 2	< 2	< 1	< 2
Back half	a	140	< 6	< 6	< 8

^a Samples from Run 1 were not analyzed because this run occurred prior to installation of a flow straightener in the stack.

TABLE G-9. STACK OUTPUT RATES FOR SEMIVOLATILE PICs
(NOT BLANK CORRECTED), g/min

	Run 1	Run 2	Run 3	Run 5	Run 4
Naphthalene					
Front half	a	< 0.00001	< 0.00001	< 0.000006	< 0.00001
Back half	a	0.013	0.0014	0.0040	0.0091
O-Nitrophenol					
Front half	a	< 0.0001	< 0.0001	< 0.00006	< 0.0001
Back half	a	0.0086	< 0.0003	< 0.0003	< 0.0005

^a Samples from Run 1 were not analyzed because this run occurred prior to installation of a flow straightener in the stack.

TABLE G-10. STACK OUTPUT VOLATILE PIC CONCENTRATIONS (NOT BLANK CORRECTED), ng/L

	Gas bags					VOST ^a				
	Aniline					Mono-nitrobenzene Run 4	Aniline			
	Run 1	Run 2	Run 3	Run 5	Run 1		3rd Pair	Run 2 ^b		
					1st Pair			2nd Pair	1st Pair	2nd Pair
Chloroform	1.1	30	2.8	0.4	1.2	0.50	0.46	0.88	34	2.3
Benzene	< 0.6	5.4	22	10	< 0.6	4.4	3.3	0.24	38	6.9
Toluene	26	24	13	22	15	10	2.4	2.7	5.2	1.4
1,1,1-Trichloroethane	0.2	2.0	< 0.2	0.6	2.4	< 0.05	1.7	0.73	0.85	0.09
Carbon tetrachloride	2.4	0.5	10	0.9	< 0.2	4.5	0.78	0.38	1.8	< 0.05
Trichloroethylene	13	7.6	56	7.4	4.4	25	5.0	4.6	16	1.8
Tetrachloroethylene	1.4	1.3	1.3	1.1	2.4	0.21	0.18	0.20	0.62	0.24
Chlorobenzene	2.0	7.4	7.2	1.6	0.50	1.8	0.79	0.73	5.5	1.2

	VOST					Mononitrobenzene						
	Aniline					Run 3	Run 4 ^b					
	1st Pair	2nd Pair	3rd Pair	Run 3			1st Pair	2nd Pair	1st Pair	2nd Pair		
				1st Pair	2nd Pair						1st Pair	2nd Pair
Chloroform	9.7	1.2	0.79	4.7	1.0	4.7	1.0	3.6				
Benzene	11	5.5	2.8	7.4	3.6	7.4	3.6	1.2				
Toluene	9.8	2.6	0.69	2.9	< 0.05	2.9	< 0.05	0.25				
1,1,1-Trichloroethane	0.07	< 0.05	0.11	0.36	0.25	0.36	0.25	1.5				
Carbon tetrachloride	1.1	1.6	< 0.05	0.63	< 0.05	0.63	< 0.05	0.60				
Trichloroethylene	8.9	2.1	1.1	4.9	1.5	4.9	1.5	0.60				
Tetrachloroethylene	0.21	0.09	< 0.05	0.16	< 0.05	0.16	< 0.05	0.60				
Chlorobenzene	0.89	0.43	0.32	1.0	0.60	1.0	0.60	0.60				

^a VOST sampling was not done in Run 5.

^b Only two pairs of traps were analyzed in Runs 2 and 4.

TABLE G-11. STACK OUTPUT RATES FOR VOLATILE PICs (NOT BLANK CORRECTED), g/min

	VOST ^a									
	Gas bags					Aniline				
	Aniline		HNB			Run 1		Run 2 ^b		
	Run 1	Run 2	Run 3	Run 5	Run 4	1st Pair	2nd Pair	1st Pair	2nd Pair	2nd Pair
Chloroform	0.000072	0.0017	0.00016	0.00002	0.000069	0.000033	0.000030	0.000058	0.0020	0.00014
Benzene	< 0.00004	0.00032	0.0012	0.00057	< 0.00003	0.00029	0.00022	0.000016	0.0023	0.00041
Toluene	0.0017	0.0014	0.00072	0.0012	0.00086	0.00066	0.00016	0.00018	0.00030	0.000082
1,1,1-Trichloroethane	< 0.000013	0.00012	< 0.000011	0.000034	0.00014	< 0.0000033	0.00011	0.000048	0.000050	0.000005
Carbon tetrachloride	0.000066	0.00030	0.00055	0.000051	< 0.000012	0.00030	0.000051	0.000025	0.00010	< 0.000003
Trichloroethylene	0.00079	0.00045	0.0031	0.00042	0.00025	0.0016	0.00033	0.00030	0.00096	0.00010
Tetrachloroethylene	0.000085	0.00077	0.00072	0.00062	0.00014	0.000014	0.000012	0.000013	0.000037	0.000014
Chlorobenzene	0.000026	0.00044	0.00040	0.00090	0.00029	0.00012	0.00052	0.000048	0.00032	0.000073

	VOST									
	Aniline					Nonnitrobenzene				
	Run 3		Run 4 ^b			Run 4 ^b		Run 5		
	1st Pair	2nd Pair	1st Pair	2nd Pair	3rd Pair	1st Pair	2nd Pair	1st Pair	2nd Pair	2nd Pair
Chloroform	0.00054	0.000068	0.000044	0.00027	0.000068	0.00027	0.00058	0.000058	0.00021	0.00021
Benzene	0.00060	0.00031	0.00016	0.00043	0.00016	0.00043	0.00021	0.000069	0.000003	0.000003
Toluene	0.00055	0.00015	0.00039	0.00017	0.00039	0.00017	0.000069	0.000014	0.000084	0.000035
1,1,1-Trichloroethane	0.000004	< 0.000003	0.000006	0.000021	0.000006	0.000021	0.000003	0.000003	0.000003	0.000003
Carbon tetrachloride	0.00061	0.000087	< 0.000003	0.00036	< 0.000003	0.00036	0.000003	0.000003	0.000003	0.000003
Trichloroethylene	0.00050	0.00012	0.00061	0.00028	0.00061	0.00028	0.000084	0.000003	0.000003	0.000003
Tetrachloroethylene	0.00012	0.000005	< 0.000003	0.000094	< 0.000003	0.000094	0.000003	0.000003	0.000003	0.000003
Chlorobenzene	0.00050	0.000024	0.00018	0.00058	0.00018	0.00058	0.000035	0.000003	0.000003	0.000003

^a VOST sampling was not done in Run 5.^b Only two pairs of traps were analyzed in Runs 2 and 4.

TABLE G-12. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>PICs</u>				
Chloroform	0.44	0.61	2.0	3.8
Benzene	12	9.5	9.4	13
Toluene	79	67	12	7.2
1,1,1-Trichloroethane	2.6	4.2	7.3	4.9
Carbon tetrachloride	1.0	2.3	< 1	0
Trichloroethylene	28	19	3.5	1.8
Tetrachloroethylene	7.3	3.8	1.8	2.5
Chlorobenzene	0.86	0.89	4.6	2.9

TABLE G-13. VOLATILE SAMPLE VOLUMES (DRY
STANDARD LITERS)

Gas bags		VOST		
Run No.	Volume (L)	Run No.	Pair No.	Volume (L)
1	5.0	1	1	19.1
2	5.0	1	2	19.5
3	5.0	1	3	14.8
4	5.0	2	1	20.1
5	5.0	2	2	20.3
		3	1	20.2
		3	2	20.6
		3	3	20.2
		4	1	19.1
		4	2	19.0

TABLE G-14. SEMIVOLATILE BLANK
CORRECTION VALUES

Compound	Blank correction value (μg)
<u>POHCs</u>	
Aniline	< 1
Phenylenediamine	< 1
Diphenylamine	< 1
Mononitrobenzene	< 1
m-Dinitrobenzene	< 5
<u>PICs</u>	
Naphthalene	16
o-Nitrophenol	< 2

TABLE G-15. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs) FOR THE AMERICAN CYANAMID INCINERATOR (BLANK CORRECTED)

Semivolatile compounds	Aniline waste		Mononitrobenzene waste
	Run 2	Run 3	Run 4
Aniline	> 99.999989	> 99.999992	> 99.9997
Phenylene diamine	> 99.997	> 99.9992	a
Diphenyl amine	> 99.999	> 99.9992	a
Mononitrobenzene	a	a	99.99991
m-Dinitrobenzene	a	a	> 99.99

a Waste feed concentration < 100 µg/g in this run.

TABLE G-16. PARTICULATE CONCENTRATION AND EMISSION RATE DATA

Particulate concentration					Mono- nitro- benzene
Aniline					
Run 1 ^a	Run 2 ^b	Run 3	Run 5	Run 4	
Measured					
mg/dscm	56.9	77.0	71.7	188.8	7.6
(gr/dscf)	(0.0249)	(0.0336)	(0.0313)	(0.0825)	(0.0033)
Corrected to 7% O ₂					
mg/dscm	92.6	171.1	157	400	15.0
(gr/dscf)	(0.0405)	(0.0746)	(0.0686)	(0.1750)	(0.0066)
Particulate emissions					
Run 1 ^a	Run 2 ^b	Run 3	Run 5	Run 4	
Emission rate					
kg/hr	0.22	0.27	0.24	0.64	0.026
(lb/hr)	(0.48)	(0.60)	(0.53)	(1.40)	(0.057)

^a Data from Run 1 may not be valid because stack gas flow was cyclonic. Flow straighteners were installed after Run 1.

^b Data for Run 2 is based on only one of the two sampling trains that traversed each stack diameter. The probe liner broke in the other train during the run.

TABLE G-17. METHOD 5 TEST DATA

	Aniline				Mono-nitro-benzene
	Run 1 ^a	Run 2	Run 3	Run 5	Run 4
Volume of gas sampled, Nm ³ ^b (dscf)	1.995 (70.44)	1.035 ^d (36.54)	1.726 (60.95)	1.745 (61.62)	1.198 (42.30)
Sampling time, min ^c	66	66	66	66	44
Percent isokinetic					
Train A	98.4	99.2	101.3	101.4	101.4
Train B	97.7	-	99.0	99.0	100.0
Moisture content, %					
Train A	5.2	3.5	4.3	6.2	5.4
Train B	4.0	-	3.2	2.9	4.9
Percent O ₂ (dry)	12.4	14.7	14.6	14.4	13.7
Percent CO ₂ (dry)	6.6	4.9	5.0	5.2	6.7
Stack gas temp., °C					
Train A	744	469	539	604	616
(°F)	(1372)	(877)	(1001)	(1120)	(1140)
Train B	654	617	612	606	665
(°F)	(1209)	(1142)	(1134)	(1122)	(1228)
Avg. gas flow rate, act. m ³ /min (act. ft ³ /min)	232 (8190)	170 (6000)	170 (6000)	180 (6360)	192 (6780)
Avg. gas flow rate, Nm ³ /min (dscf/min)	66 (2330)	59 (2080)	56 (1980)	57 (2010)	58 (2050)

^a Run 1 was conducted prior to installation of a flow straightener in stack.

^b Total volume of gas sampled by both Trains A and B.

^c Sampling time for each train.

^d Train A only (probe liner broken in Train B during Run 2).

TABLE G-18. CONTINUOUS MONITORING DATA^a

	Aniline			Mono-nitro-benzene Run 4
	Run 1	Run 2	Run 3	Run 5
Oxygen (%)				
Range	9.1-13.4	7.1-15.2	c	10.3-14.6
Average	10.3	12.4	c	13.0
Carbon dioxide (%)				
Range	5.8-9.8	5.5-10.7	c	5.2-7.5
Average	8.8	6.8	c	6.1
Carbon monoxide (ppm)				
Range	2.1-37.7	7.2-377	c	6.4-44
Average	4.0	30.6	c	14.3
Total hydrocarbon (ppm) ^b				
Range	< 1-1.8	< 1-1.6	< 1-1.2	< 1-1.1
Average	< 1	< 1	< 1	< 1

^a Concentrations on dry gas basis.

^b Total HC as propane.

^c Electrical problem shut down all monitors except total HC.

TABLE G-19. CHLORIDE EMISSIONS

	Aniline				Mono-nitro-benzene
	Run 1	Run 2	Run 3	Run 5	Run 4
<u>Chloride emissions</u>					
kg/hr	0.004	0.002	0.003	0.003	0.003
(lb/hr)	(0.01)	(0.004)	(0.007)	(0.007)	(0.007)

ANALYSIS OF WASTE FEEDS

Table G-20 summarizes data on percent chlorine, percent H₂O, percent ash, and heating value for the waste feeds. The aniline waste data show little variation between test runs.

METALS

The concentration of metals in the waste feed and particulate emissions was determined for Run 3. In addition to the 12 hazardous metals listed in the Guidance Manual for Hazardous Waste Incineration Permits, the concentrations of six other metals are reported in Table G-21 in order to further define the composition of the particulate. Analysis shows that the particulate contained about 22% iron. Chromium and nickel were the only hazardous metals found in the waste feed at a concentration of > 5 µg/g, and were the only hazardous metals present at a concentration of > 20,000 µg/g in the particulate.

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (1) external auditing; (2) sampling QA activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. A comprehensive performance audit (quantitative evaluation) and limited systems audit (qualitative evaluation) were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on:

- GC/MS analysis of volatile and/or semivolatile organics; and
- Technicon Autoanalyzer analysis of chlorides.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics and semivolatile organics. This portion of the analytical program is judged to be within control limits.

Analyses of chloride audit samples were determined to be adequate and acceptable. This part of the analytical phase is also within control limits.

Systems audits demonstrated that laboratory analyses were done according to protocol.

TABLE G-20. GENERAL ANALYSIS OF WASTE FEED

	Percent Cl	Percent water	Percent ash	Heating value, kJ/kg (Btu/lb)
<u>Aniline waste</u>				
Run 1	0.018	6.02	0.16	33,570 (14,500)
Run 2	0.015	5.22	0.19	33,620 (14,522)
Run 3	0.020	5.48	0.19	33,544 (14,490)
Run 5	0.019	7.26	0.50	33,490 (14,465)
<u>Mononitrobenzene waste</u>				
Run 4	0.013	0.57	< 0.05	24,960 (10,784)

TABLE G-21. CONCENTRATION OF METALS IN WASTE FEED
AND PARTICULATE EMISSIONS (RUN 3)^a

	Conc. in waste feed (µg/g)	Conc. in particulate (µg/g)	
		Train A	Train B
Arsenic	11	b	b
Silver	< 0.45	930	310
Beryllium	< 0.5	b	b
Barium	< 0.24	< 1,000	< 1,000
Cadmium	< 0.84	< 1,200	< 1,200
Chromium	250	47,000	48,000
Nickel	170	47,000	51,000
Lead	1	< 4,000 ^c	< 4,000 ^c
Antimony	< 2	< 12,000 ^c	< 12,000 ^c
Selenium	10	b	b
Thallium	5	< 10,000 ^c	< 10,000 ^c
Mercury	4	< 16,000 ^c	< 16,000 ^c

Other metals detected in samples^d

Copper	1	12,000	13,000
Iron	1,300	222,000	230,000
Manganese	34	8,000	8,300
Molybdenum	4	1,600	1,800
Sodium	71	10,000	10,000
Zinc	43	1,300	1,500

^a Only samples from Run 3 were analyzed for metals.

^b Values are less than or about the same as blank values.

^c Detection limits are relatively high for some metals due to low ratio of particulate weight to filter weight and amounts in blank filter.

^d Other metals are reported due to interest in composition of particulate.

Sampling QA Activities

In general, sampling QA activities are scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures included spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure used was monitoring the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX H

SUMMARY OF RESULTS FROM TESTING AT THE
MITCHELL SYSTEMS INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration which requires the collection of data on current incineration operating practices. Through a comprehensive sampling and analysis program at selected incineration facilities, test data regarding water feed composition, incinerator effluents and operating parameters are being acquired.

The EPA selected the hazardous waste incinerator at Mitchell Systems, Inc. in Spruce Pine, North Carolina as one facility for the test program. On-site sampling was conducted on November 3-5, 1982. Samples of the liquid waste feeds and the stack effluent were collected and analyzed. The following sections of this appendix present a summary of the emission measurement results, a brief process description, a synopsis of the test plan and the sampling and analysis methods used, and a discussion of the test program results.

SECTION 2

SUMMARY OF RESULTS

The major emphasis of the Mitchell Systems test program was the determination of principal organic hazardous constituents (POHCs) in the waste feed and the stack effluent. The compounds shown in Table H-1 were identified either as POHCs (i.e., greater than 100 $\mu\text{g/g}$ in the waste materials), or were cited as products of incomplete combustion (PICs) in the stack effluent (not found in the waste feeds).

Table H-2 presents a summary of the emissions of several major (non-organic) components of the stack effluent. The results shown are averages of the data from three sampling runs. Since the incinerator was not equipped with a scrubber or other pollution control device, the results in Table H-2 represent uncontrolled emissions.

Particulate emissions averaged 903 mg/dscm (0.394 gr/dscf) when corrected to 7% O_2 , a value greater than the federal particulate emission limitation of 180 mg/dscm (0.08 gr/dscf) for hazardous waste incinerators.

Chloride emissions averaged 32.3 g/min (4.3 lb/hr). Although the federal limitation for chloride emissions from hazardous waste incinerators of 4 lb/hr was exceeded, it must be noted that essentially all chlorinated materials in the waste feed to the incinerator were added for this test. Carbon tetrachloride and trichloroethylene were injected into the waste feed line during each run.

All of the Mitchell Systems test results, including calculated DREs, are presented in detail in Section 5. This appendix contains minimal interpretation of the results.

TABLE H-1. SUMMARY OF POHCs AND PICs IDENTIFIED
AT MITCHELL SYSTEMS

POHCs	PICs
<u>Volatile compounds</u>	
Carbon tetrachloride	Methylene chloride
Trichloroethylene	Chloroform
Benzene	1,1,1-Trichloroethane
Methyl ethyl ketone	Chlorobenzene
Tetrachloroethylene	
Toluene	
<u>Semivolatile compounds</u>	
Phenol	2,4-Dimethylphenol
Naphthalene	
Butylbenzylphthalate	
Bis(2-ethylhexyl)phthalate	

TABLE H-2. AVERAGE STACK EFFLUENT
SAMPLING RESULTS

<u>Particulate emissions</u>	
mg/dscm corrected to 7% O ₂	903
gr/dscf corrected to 7% O ₂	0.394
<u>Chloride emissions^a</u>	
g/min	32.3
lb/hr	4.3

^a All chlorinated waste feed materials were spiked into the waste feed for this test.

SECTION 3

PROCESS DESCRIPTION AND OPERATING DATA

The incinerator tested is a commercial facility designed primarily to burn liquid wastes, but also with the capability to accept solid wastes in small quantities. The unit consists of two, horizontally-oriented, cylindrical combustion chambers (primary and secondary) situated one above the other. Combustion gases exit through a stack at one end of the secondary chamber. A schematic diagram of the system is shown in Figure H-1, and a drawing of the combustion chambers, including thermocouple (T/C) locations, is presented in Figure H-2.

Liquid wastes are delivered to the plant in tank trucks and stored in one of four main storage tanks (20,000 gal. each). The wastes can then be transferred to two, 12,500 gal. blend tanks equipped with mixers and level gauges before they are fed to the incinerator. Two types of liquid wastes, organic and aqueous, are normally processed by the facility. They are stored and handled separately. Waste chemicals are also received at the plant in 55 gal. drums which are stored inside the building. When scheduled for incineration, the drums are emptied into a small holding tank that is equipped with an exhaust hood. The liquid wastes can then be pumped from the base of the tank directly to the incinerator. Capacity of the tank is approximately 630 gal.

Waste liquids are fed to the primary combustion chamber using two, air-atomized injectors (a total of four are available). Flow rates are established by adjusting manually operated valves. The primary combustion chamber is approximately 10 ft. long and has an outside diameter of 8 ft. The firing end has an opening about 60 in. wide with a variable height depending upon the position of an adjustable shutter which is used to regulate the natural draft of combustion air. The unit is rated at 9.5×10^6 Btu/hr, with a normal operating temperature of 1800 to 2100°F in both chambers. The secondary chamber is identical to the primary except that it is only 7 ft in diameter (both chambers are refractory lined). Typical retention times are 2 to 3 sec.

The exhaust stack (which is also refractory lined) extends to a height of 20 ft above the secondary combustion chamber. Combustion gases exiting the stack typically average about 1450°F. At the beginning of each week's operation, ash which has accumulated on the bottom of the primary chamber from the previous week's operation is manually removed and stored in an outdoor concrete holding bay. Eventually the ash is sent to a landfill.

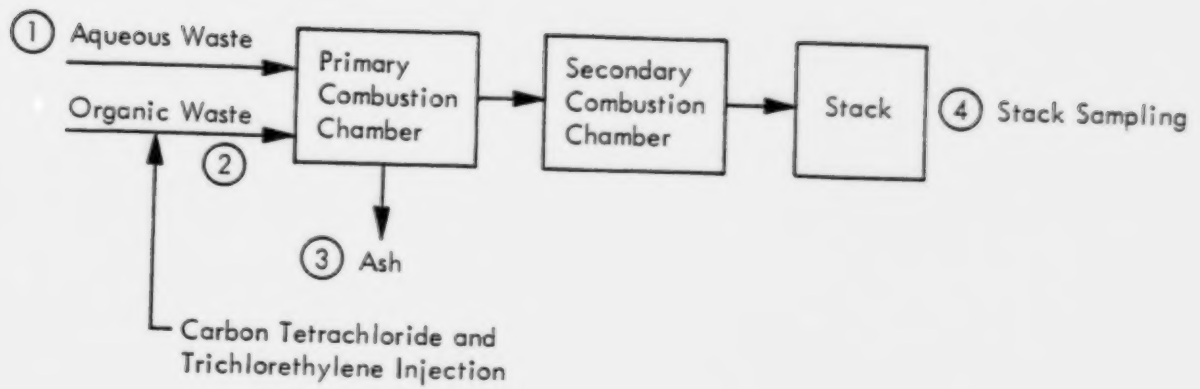
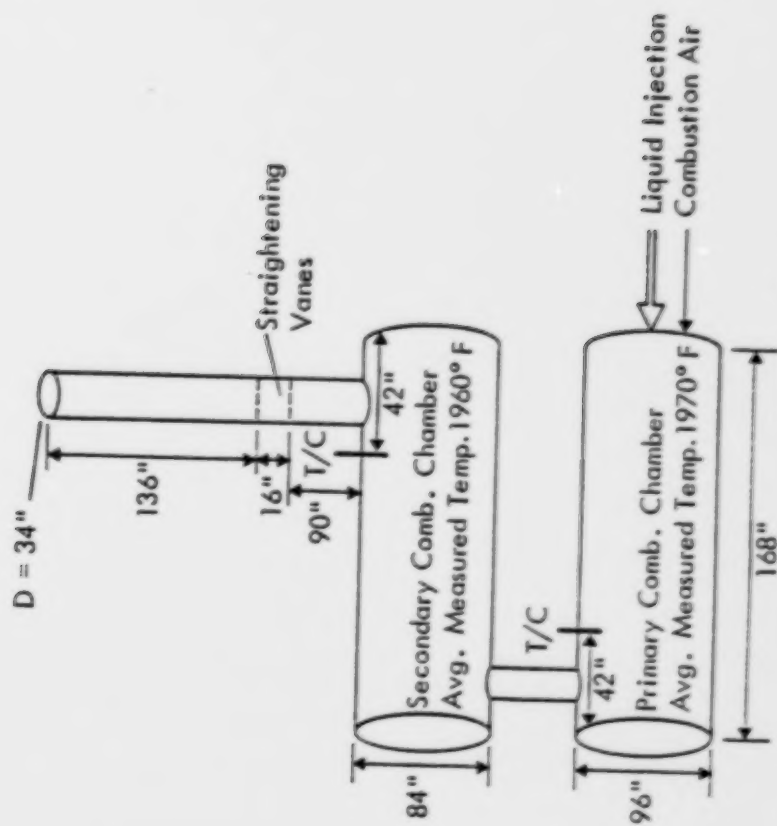


Figure H-1. Schematic diagram of incinerator with sampling locations.



Note:
T/Cs extend inside,
5-6" past refractory

Figure II-2. Combustion chamber diagram.

System operating data obtained during the tests are summarized in Table H-3 for each sampling period. The calculated heat input rate data show that the unit was operated near its rated capacity throughout the test program. Information obtained from the limited process monitoring instrumentation indicated a relatively constant incinerator operation among the four sampling runs.

TABLE H-3. INCINERATOR OPERATING CONDITIONS^a

Parameter	Run 1, 11/3/82	Run 2, 11/4/82	Run 3, 11/4/82	Run 4, 11/5/82
Organic waste flow rate, ^b kg/min (lb/hr)	3.76 (497)	4.01 (542)	4.50 (595)	4.25 (562)
Aqueous waste flow rate, ^b kg/min (lb/hr)	6.13 (811)	5.38 (712)	4.90 (648)	5.61 (742)
Heat input rate, GJ/hr (MMBtu/hr)	8.37 (7.93)	9.01 (8.54)	10.50 (9.96)	9.38 (8.89)
Combustion chamber temp., °C (°F)				
Upper	1053 (1925)	1066 (1950)	1094 (2000)	1080 (1975)
Lower	1011 (1850)	1094 (2000)	1122 (2050)	1080 (1975)
Calculated residence time, ^c sec	2.5	2.4	2.2	2.2
Stack height, ft	38	38	38	38
Stack exit velocity, fpm	2,110	2,030	2,230	2,340
Stack temperature, °F	1490	1380	1410	1490
Percent opacity, ^d range	1-3	4-8	5-10	0-4

^a Data collected by reading plant monitoring instruments at regular intervals. Values shown are averages for each run.

^b Determined by measuring storage tank liquid levels at start and finish of each run.

^c Calculated from chamber volumes and stack flow rates.

^d Opacity measured by in-stack transmissometer.

SECTION 4

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

SAMPLING METHODOLOGY

A summary of the sampling and analysis activities conducted is presented in Table H-4. It is organized according to the sampling points previously identified in Figure H-1. In addition to the comprehensive sampling of the stack effluent, samples of the two waste feeds and the incinerator residual ash were also collected.

Liquid waste samples were obtained from valves on the "Christmas tree" manifold located at the firing end of the incinerator. Flexible hoses were used to connect the pipe injectors to the proper waste feed valves. Small grab samples of each waste stream were taken every 15 min. for the duration of each sampling run and composited into a 1-L container.

A 50:50 mixture (by volume) of carbon tetrachloride (CCl_4) and trichloroethylene (TCE) was continuously pumped (spiked) into the organic waste feed line at a rate of 30 mL/min. The valve used for spiking the organic waste was located about 1 ft upstream from the valve used to collect the organic waste samples.

A single grab sample of the incinerator bottom ash was collected for analysis by Mitchell Systems personnel on the Monday morning following the tests. Because of the high ash temperatures in between sampling runs, an ash sample could not be taken until material had sufficiently cooled. Usual plant procedure is to remove the bottom ash at the start of each week's operation.

Stack sampling methods included a modified EPA Method 5 for particulates, semivolatile POHCs and PICs, and chlorides; two methods for collecting volatile POHCs and PICs, an integrated gas bag and the volatile organic sampling train (VOST); gas bag for Orsat determination of CO_2 and O_2 ; and continuous gas analyzers for O_2 , CO , CO_2 , and total hydrocarbons. A schematic diagram of the MM5 train is shown in Figure H-3, and the VOST is diagrammed in Figure H-4.

Gas bag samples were collected in 15-L Tedlar bags, drawing gas into the bags over a period of about 1 hr. The VOST train consists of a pair of Tenax and Tenax/charcoal traps (one each) in series. Sample gases are drawn through the two traps for the appropriate length of time based on flow rate. The pair of traps is then removed and replaced with another pair, until the desired total number of pairs have been used.

TABLE H-4. SUMMARY OF SAMPLING AND ANALYSIS ACTIVITIES

Sampling point	No. of samples per run	Sampling technique	Analysis
<u>Waste feeds</u>			
1. Aqueous waste	1 composite	Grab sample every 15 min.	POHCs, HHV, Cl ⁻ , %H ₂ O, % ash, viscosity
2. Organic waste	1 composite	Grab sample every 15 min.	Same as above and ultimate
<u>Bottom ash</u>			
3. Primary chamber - One grab sample collected by plant personnel on Monday following test week			POHCs, Cl ⁻
<u>Combustion effluent</u>			
4. Stack	1	Modified EPA Method 5	Particulate weight, semivolatiles, POHCs and PICs, Cl ⁻
	1 (6 pairs of traps)	VOST (fast flow rate)	Volatiles POHCs & PICs
	1 (3 pairs of traps)	VOST (slow flow rate)	Volatiles POHCs & PICs
	1	Integrated gas bag	Volatiles POHCs & PICs
	1	Integrated gas bag	Orsat (CO ₂ , O ₂)
	Continuous	Continuous gas analyzers	CO, CO ₂ , O ₂ , THC

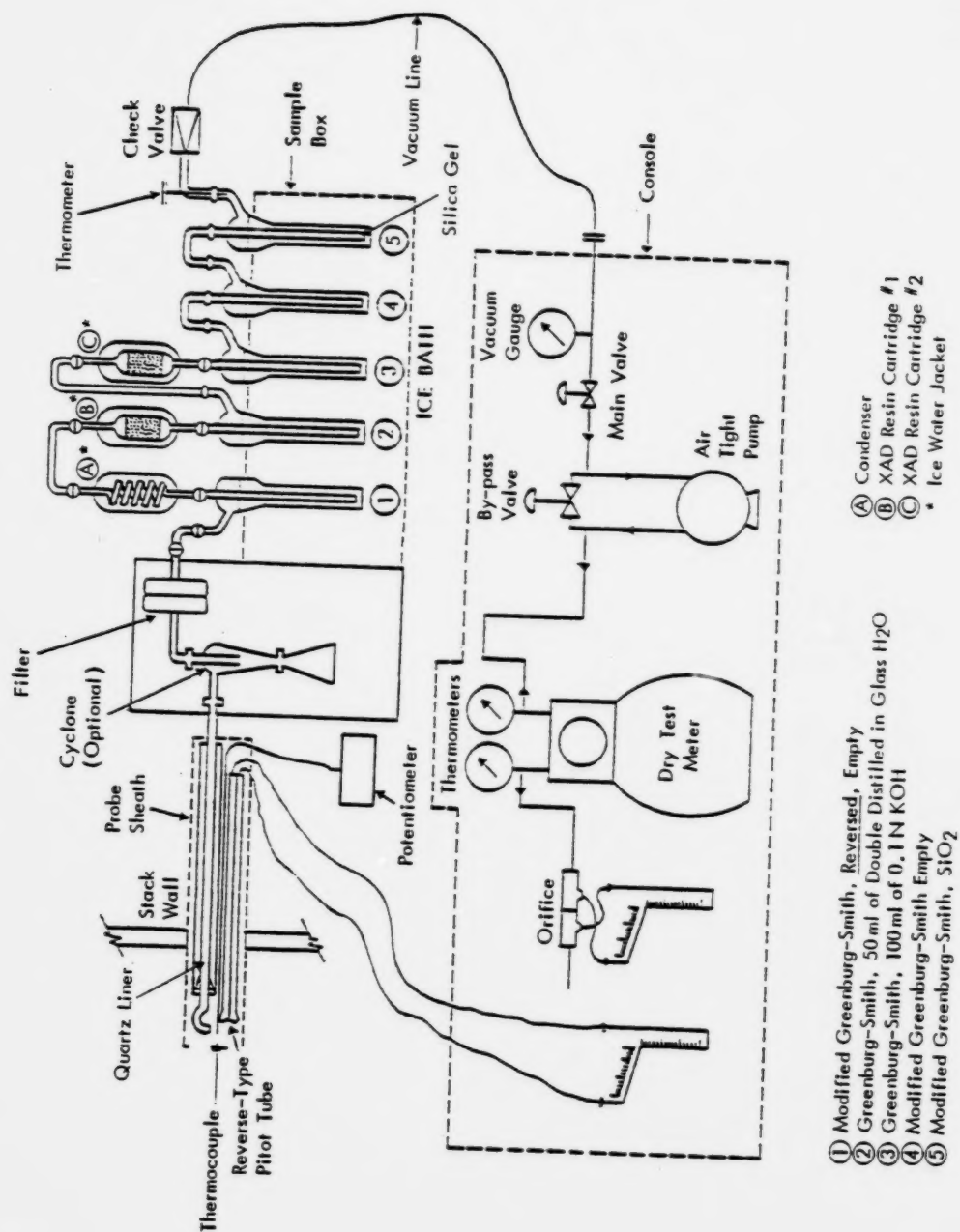
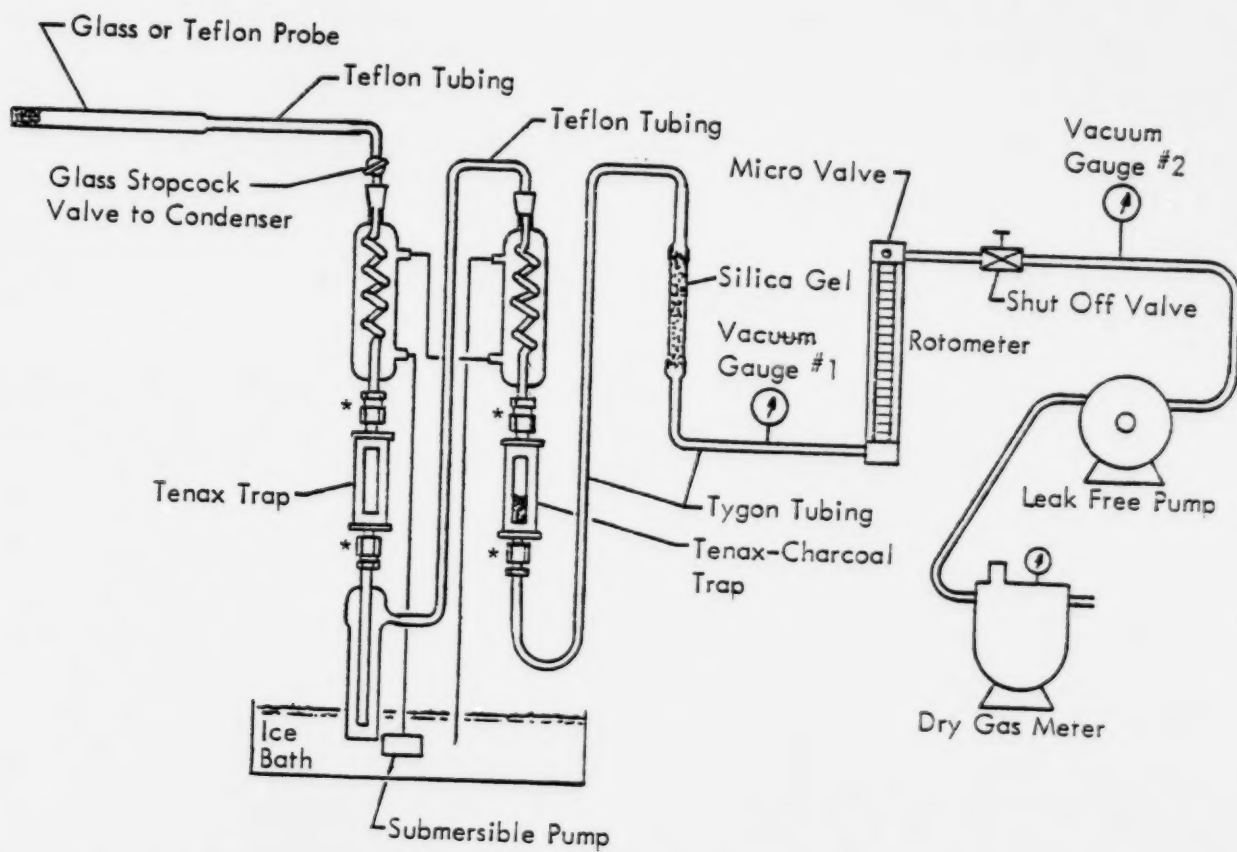


Figure II-3. Modified Method 5 sampling train (MM5).



* Special O-Ringed Stainless Steel Fittings

Figure H-4. Volatile organic sampling train (VOST).

For this site-specific test program, VOST samples were collected at two different flow rates. The fast VOST samples were collected at a rate of 1 L/min. Each pair of traps was inserted in the train for 20 min and a total of six pairs were used (120 min total). Slow VOST samples were collected at a rate of 0.25 L/min, using three pairs of traps for 20 min each. The traps and the gas bags were returned to the analytical laboratory for analysis of volatile POHCs.

WASTE FEED ANALYSIS

Replicates of the liquid waste feed samples were collected for determination of higher heating value and chloride content. The remaining waste feed analyses for POHCs were done using GC/MS techniques. Prior to analysis, the samples were homogenized either manually or mechanically depending upon their physical state. The homogenized feed samples were analyzed for volatile POHCs using a method based on Method A101b in the A. D. Little report entitled "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0 g aliquot of the mixed waste feed sample was dispersed in 40 mL of prepurified tetraglyme. A 10- μ L aliquot of the tetraglyme solution was spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample was then analyzed by the purge-trap-desorb technique as described in Method A101A in the A. D. Little report (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis may be repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs were analyzed in the waste feed samples according to Method P023 in the A. D. Little report. A 5-g portion of the homogenized waste feed sample was spiked with semivolatile surrogate compounds, such as decafluorobiphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 μ g/g depending on the expected sample complexity. The sample was allowed to equilibrate with the spiked compounds and then was extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appeared on the surface of the methylene chloride, the pH was checked and the sample pH neutralized. The extract was then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

An aliquot of the extract was then screened by gas chromatography with a flame ionization detector. If necessary, the extract was further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column (FSCC). A 1.0-mL aliquot of the extract was then spiked with 20 μ g of D₁₀-anthracene internal standard and analyzed by GSCC GC/MS.

Heating value of the waste feed samples was determined according to ASTM Method D 240-64. Chloride content was determined by potentiometric titration using a Fisher Titralyzer Model 41. Percent water content was measured using the Karl Fisher method and percent ash was determined by ASTM Method D 482. The viscosity of the samples was determined by the Saybolt Method (ASTM D 88).

BOTTOM ASH ANALYSIS

The grab sample of the incinerator bottom ash was analyzed for POHCs and chlorides using the same methods outlined in the previous section for the liquid waste samples.

STACK EFFLUENT ANALYSIS

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with the VOST. A known amount of the gas bag sample, usually 5.0 L was transferred onto a Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). A needle was inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were analyzed using the same techniques as for the VOST traps described below.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene (BFB) internal standards using the flash vaporization technique in which the spiking solution was vaporized and carried onto the trap with a carrier gas. The glass traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, was turned on through the trap and a syringe needle injected through the GC septum port. One microliter of a 25 ng/μl methanolic solution of the internal standards was slowly expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL) which is a component of the EPA Method 24 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds passed into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs in the stack samples involves several components of the MMS train. Each of the components of the MMS train were spiked with a separate surrogate standard according to the following scheme:

Front XAD-2 resin trap: 20 μg of ¹³C-tetrachlorobiphenyl
Back XAD-2 resin trap: 20 μg of decafluorobiphenyl
Particulate filter plus probe rinse residue: 20 μg of D₁₂-chrysene
Condensate water: 20 μg of D₈-naphthalene

The spiked components were normally extracted separately, and then the extracts were combined, dried with anhydrous sodium sulfate, concentrated to 1.0 mL using Kuderna-Danish evaporation according to Method P031 in the

A. D. Little report and using supplementary gentle nitrogen blowdown, and analyzed by FSCC GC/MS. The samples were extracted according to the following procedures:

XAD-2 Resin Traps

The XAD-2 resin traps were Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Particulate Filters Plus Probe Rinse Residues

The residue from the acetone rinse of the probe was added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Condensate Water

The condensate water was extracted with three 60-mL aliquots of methylene chloride as described in Method P021a in the A. D. Little report (equivalent to EPA Method 625).

EPA Method 3 was followed when conducting Orsat analysis of the stack effluent. The continuous monitoring instrumentation consisted of the following:

- O₂ - Beckman Model 742 (polarographic sensor)
- CO - Beckman Model 215A (nondispersive infrared)
- HC - Beckman Model 402 (heated oven/flame ionization detector)
- CO₂ - Horiba Model PIR-2000S (nondispersive infrared)

More detailed descriptions of all the above sampling and analysis methods are presented in Appendix B.

SECTION 5

PRESENTATION OF RESULTS

VOLATILE AND SEMIVOLATILE POHCs

Input and Output Concentrations and Rates for POHCs

POHC input and output concentrations and mass rates were determined for all POHCs listed by EPA which were detected in the waste feeds at concentrations above 100 µg/g. No attempt was made to restrict the number of POHCs based on any selection or ranking method. The POHCs reported were quantified against authentic standards whenever possible. Where authentic standards were not available, estimated GC/MS response factors were used to provide semiquantified results. In all tables, values marked < indicate detection levels.

The concentrations of both volatile and semivolatile POHCs measured in the two waste feed samples are shown in Table H-5. The same results, expressed as input rates (g/min), are presented in Table H-6 and were based on the waste feed rates previously presented in Table H-3. The data in Table H-5 indicate that the organic waste feed contained mainly methyl ethyl ketone, toluene, phenol and bis(2-ethylhexyl)-phthalate, as well as the two compounds spiked into the waste (carbon tetrachloride and trichloroethylene). The primary POHC in the aqueous waste was phenol.

POHC emission data are presented in Tables H-7 through H-9. Volatile POHC results are shown for both the gas bag and the VOST sampling methods, while the semivolatile results were obtained from the modified Method 5 samples. Table H-7 lists the output concentrations in units of nanograms per liter of stack gas (not blank corrected). The calculated POHC output rates are shown in Table H-8.

Semivolatile POHC results, presented in Table H-9, showed generally low concentrations of the four compounds identified in the waste feed. Isophorone is not listed as an emitted semivolatile POHC, even though it was identified as a POHC in the waste, because of an analytical interference caused by the acetone used to rinse the Modified Method 5 sampling probe. The most prevalent compound of the group was bis(2-ethylhexyl)-phthalate.

Blank Values

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table H-10.

TABLE H-5. CONCENTRATIONS OF POHCs IN WASTE FEEDS (µg/g)

	Aqueous waste				Organic waste			
	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4
Volatile POHCs								
Carbon tetrachloride	< 2 ^a	< 2	< 2	< 2	6,400	6,000	4,700	5,600
Trichloroethylene	< 1	< 1	< 1	< 1	5,900	5,500	4,300	5,200
Benzene	< 3	< 3	< 3	< 3	2.7	260	140	85
Toluene	94	110	100	98	1,800	2,400	1,900	1,300
Methyl ethyl ketone	14	27	30	34	7,200	9,700	7,300	6,500
Tetrachloroethylene	1.3	< 1	< 1	< 1	15	28	180	50
Semivolatiles POHCs								
Phenol	42,000	34,000	b	30,000	4,200	1,000	b	260
Isophorone	< 100	< 100	b	< 100	< 100	110	b	500
Naphthalene	< 100	< 100	b	< 100	510	350	b	920
Butylbenzylphthalate	< 100	< 100	b	< 100	200	320	b	150
Bis(2-ethylhexyl)phthalate	< 100	< 100	b	< 100	5,000	3,800	b	9,600

^a Results reported as less-than values represent limits of detection.

^b MM5 sample voided for this run due to equipment problems. Therefore, the waste feed samples were not analyzed for semivolatiles POHCs.

TABLE H-6. CALCULATED INPUT RATES FOR POHCs
IN WASTE FEEDS

	Input rates (g/min) ^a			
	Run 1	Run 2	Run 3	Run 4
<u>Volatile POHCs</u>				
Carbon tetrachloride	24	25	21	24
Trichloroethylene	22	22	19	22
Benzene	0.010	1.1	0.63	0.36
Toluene	7.3	10	9.0	6.1
Methyl ethyl ketone	27	40	33	28
Tetrachloroethylene	0.064	0.012	0.81	0.21
<u>Semivolatile POHCs</u>				
Phenol	270	180	b	170
Isophorone	< 1	0.45	b	2.1
Naphthalene	1.9	1.4	b	3.9
Butylbenzylphthalate	0.75	1.3	b	0.64
Bis(2-ethylhexyl)phthalate	19	16	b	41

^a Combined input rates for both waste feeds.

^b Samples not analyzed for semivolatiles since MM5 sample was voided for this run.

TABLE H-7. CONCENTRATIONS OF VOLATILE POHCs IN STACK EFFLUENT (NOT BLANK CORRECTED), ng/L

	Run 1								
	Fast VOST ^a				Slow VOST ^a				Gas bag
	1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair ^d	3rd pair	Avg.	
Carbon tetrachloride	2.3	0.47	0.57	1.1	24	3.7	1.3	9.7	10
Trichloroethylene	20	1.8	1.6	7.8	120	8.1	7.7	45	110
Benzene	2.2	2.3	2.2	2.2	12	2.9	6.7	7.2	7.6
Tetrachloroethylene	< 0.2	< 0.06	0.097	< 0.1	1.8	< 0.2	< 0.1	< 0.7	2.0
Toluene	6.2	0.99	2.1	3.1	55	46	6.5	36	66
Methyl ethyl ketone	1.6	1.4	1.8	1.6	35	5.0	4.7	14	22

	Run 2								
	Fast VOST ^a				Slow VOST ^a				Gas bag
	1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	3rd pair	Avg.	
Carbon tetrachloride	2.3	1.7	1.7	1.9	61	c	6.5	33	7.2
Trichloroethylene	17	1.8	1.0	6.6	41	c	12	27	84
Benzene	2.0	7.4	2.6	4.0	12	c	29	16	3.4
Tetrachloroethylene	< 0.05	0.065	< 0.2	< 0.09	0.33	c	< 0.2	< 0.3	1.2
Toluene	21	7.5	4.3	11	120	c	40	80	26
Methyl ethyl ketone	9.3	3.6	2.1	5.0	47	c	6.9	27	15

	Run 3									
	Fast VOST ^a				Slow VOST ^a				Gas bag	
	1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	3rd pair	Avg.	Run 3	Run 4
Carbon tetrachloride	3.1	0.58	0.45	1.4	64	42	13	40	11	6.4
Trichloroethylene	4.8	0.95	0.66	2.1	17	5.3	5.1	9.1	56	42
Benzene	6.0	7.1	6.2	6.4	14	14	13	14	8.6	7.8
Tetrachloroethylene	0.17	0.18	< 0.1	< 0.2	1.1	< 0.2	0.69	< 0.7	1.6	1.0
Toluene	15	9.7	5.7	10	92	28	29	50	18	22
Methyl ethyl ketone	11	9.1	6.0	8.7	80	43	27	50	22	46

^a The 1st, 3rd, and 6th pairs of the fast VOST correspond to the 1st, 2nd, and 3rd pairs of the slow VOST, respectively.

^b Average of 1st and 3rd pairs.

^c Data from this sample were lost during analysis.

^d Approximately 19 L of sample (versus 5 L for other slow VOST pairs) was inadvertently drawn through this pair.

TABLE B-8. OUTPUT RATES FOR VOLATILE POHCS IN STACK EFFLUENT (NOT BLANK CORRECTED), g/min

Run 1									
Fast VOST ^a					Slow VOST ^a				
1st pair	3rd pair	6th pair	Avg.		1st pair	2nd pair ^d	3rd pair	Avg.	Gas bag
Carbon tetrachloride	0.00017	0.000034	0.000042	0.000080	0.0018	0.00027	0.000095	0.00071	0.00074
Trichloroethylene	0.015	0.00013	0.00012	0.00057	0.0088	0.00059	0.00056	0.0033	0.0077
Benzene	0.00016	0.00017	0.00016	0.00016	0.00088	0.00021	0.00049	0.00053	0.00055
Tetrachloroethylene	< 0.00001	0.0000040	0.0000071	< 0.0000008	0.00013	< 0.00001	< 0.00001	< 0.00005	0.00015
Toluene	0.00045	0.000072	0.00015	0.00023	0.0040	0.0034	0.00047	0.0026	0.0048
Methyl ethyl ketone	0.00012	0.00010	0.00013	0.00012	0.0024	0.00037	0.00034	0.0010	0.0016

Run 2									
Fast VOST ^a					Slow VOST ^a				
1st pair	3rd pair	6th pair	Avg.		1st pair	2nd pair	3rd pair	Avg. ^b	Gas bag
Carbon tetrachloride	0.00017	0.00013	0.00013	0.00014	0.0046	c	0.00049	0.0025	0.00055
Trichloroethylene	0.0013	0.00014	0.000076	0.00050	0.0031	c	0.00091	0.0021	0.0064
Benzene	0.00015	0.00056	0.00020	0.00030	0.00091	c	0.0022	0.0012	0.0026
Tetrachloroethylene	< 0.000004	< 0.00001	< 0.000007	0.000025	0.00025	c	< 0.00002	< 0.00002	0.000088
Toluene	0.0016	0.00057	0.00013	0.00084	0.0091	c	0.0030	0.0061	0.0020
Methyl ethyl ketone	0.00071	0.00027	0.00016	0.00038	0.0040	c	0.00052	0.0021	0.0011

Run 3									
Fast VOST					Slow VOST				
1st pair	3rd pair	6th pair	Avg.		1st pair	2nd pair	3rd pair	Avg.	Gas bag
Carbon tetrachloride	0.00026	0.000049	0.000038	0.00012	0.0054	0.0035	0.0011	0.0034	0.00091
Trichloroethylene	0.00040	0.000080	0.000055	0.00018	0.0014	0.00045	0.00043	0.00076	0.00054
Benzene	0.00050	0.00060	0.00052	0.00054	0.0012	0.0012	0.0011	0.0012	0.0035
Tetrachloroethylene	0.000014	0.000015	< 0.00001	< 0.00001	0.000092	< 0.00002	0.000058	< 0.00006	0.00072
Toluene	0.0013	0.00081	0.00048	0.00084	0.0077	0.0024	0.0024	0.0042	0.00066
Methyl ethyl ketone	0.00092	0.00076	0.00050	0.00073	0.0067	0.0036	0.0023	0.0042	0.00087

^a The 1st, 3rd, and 6th pairs of the fast VOST correspond to the 1st, 2nd, and 3rd pairs of the slow VOST, respectively.

^b Average of 1st and 3rd pairs.

^c Data from this sample were lost during analysis.

^d Approximately 19 L of sample (versus 5 L for other slow VOST pairs) was inadvertently drawn through this pair.

TABLE H-9. EMISSIONS OF SEMIVOLATILE POHCs
(NOT BLANK CORRECTED)

Compound	Emission concentration (ng/L)		
	Run 1	Run 2	Run 4
Phenol			
Front	< 0.4	< 0.5	< 0.6
Back	56	< 0.46	7.4
Naphthalene			
Front	< 0.1	< 0.1	< 0.2
Back	14	6.7	9.2
Butylbenzylphthalate			
Front	< 0.4	0.42	0.57
Back	< 0.4	< 0.4	1.4
Bis(2-ethylhexyl)phthalate			
Front	2.1	15	15
Back	3.4	2.1	4.2

Compound	Output rate (g/min)		
	Run 1	Run 2	Run 4
Phenol			
Front	< 0.00003	< 0.00004	< 0.00005
Back	0.0041	< 0.00004	0.00062
Naphthalene			
Front	< 0.00001	< 0.00001	< 0.00001
Back	0.0010	0.00051	0.00077
Butylbenzylphthalate			
Front	< 0.00003	0.000032	0.000048
Back	< 0.00003	< 0.00003	0.00012
Bis(2-ethylhexyl)phthalate			
Front	0.00015	0.0011	0.0013
Back	0.00025	0.00016	0.00035

Note: Isophorone is not shown as a semivolatile POHC even though it was identified as a POHC in the waste feed due to an analytical interference caused by the acetone used to rinse the Modified Method 5 sampling probe.

TABLE H-10. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>				
Carbon tetrachloride	9.3	6.8	< 2	0
Trichloroethylene	150	100	< 1	0
Benzene	130	160	< 3	0
Toluene	8.9	3.2	3.7	1.8
Methyl ethyl ketone	67	25	21	14
Tetrachloroethylene	15	8	7.9	9.2
<u>PICs</u>				
Methylene chloride	58	72	1.0	2.0
Chloroform	2.6	1.4	1.1	0.8
1,1,1-Trichloroethane	4.3	2.6	< 1	0
Chlorobenzene	19	16	< 1	0

The sample volumes for gas bags as well as slow and fast VOST are presented in Table H-11. Blank correction data for semivolatile POHCs and PICs are presented in Table H-12. Sample volumes for the MM5 train can be found in Table H-17.

Destruction and Removal Efficiencies

Destruction and removal efficiencies (DREs) based on the above data are presented in Table H-13. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$\text{DRE} = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

EMISSION OF PICs

The stack effluent sample analysis included the identification of PICs, i.e., those compounds identified in the incinerator emission samples that are listed as POHCs by EPA, but which were not measured in the waste feed samples at concentrations above 100 µg/g. Tables H-14 through H-15 present the volatile PIC results, while the semivolatile PIC results are shown in Table H-16. Concentrations of all four volatile PICs were relatively low and within the same range.

Only one semivolatile PIC could be detected in samples from the three runs, 2,4-dimethylphenol. It was detected only in the back XAD cartridge in the Modified Method 5 train used for Run 2. All other samples and blanks contained no detectable levels of the compound.

STACK EFFLUENT MEASUREMENTS

Particulate Emissions

Particulate emission results from the modified EPA Method 5 tests are presented in Table H-17. Other significant test parameters measured during the MM5 runs are also included for comparison. Most parameters were consistent among the three runs. It should be noted that Run 3 had to be voided after it was discovered that the probe liner had broken during the run and the fritted glass filter support broke during the post-test leak check.

The data in Table H-17 show that the particulate emissions from the incinerator were in excess of the federal limitation of 180 mg/dscm (0.08 gr/dscf) when corrected to 7% O₂. The lowest measured concentration was 719 mg/dscm, and the highest was 1,125 mg/dscm.

TABLE H-11. VOLATILE SAMPLE VOLUMES
(DRY STANDARD LITERS)

Gas bags		VOST				
Run No.	Volume (L)	Run No.	Fast		Slow	
			Pair No.	Volume (L)	Pair No.	Volume (L)
1	5.0	1	1	18.4	1	4.3
2	5.0	1	3	18.1	2	19.1
3	5.0	1	6	17.5	3	7.9
4	5.0	2	1	18.4	1	4.9
		2	3	18.5	2	4.9
		2	6	18.5	3	4.9
		3	1	18.9	1	4.9
		3	3	18.9	2	4.9
		3	6	19.0	3	4.9

TABLE H-12. SEMIVOLATILE BLANK
CORRECTION VALUES

Compound	Blank correction value (μg)
<u>POHCs</u>	
Phenol	3.4
Naphthalene	6.0
Butyl benzyl phthalate	< 1
Bis(2-ethylhexyl)phthalate	3.2
<u>PICs</u>	
2,4-Dimethylphenol	< 2

TABLE H-13. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs) FOR THE MITCHELL INCINERATOR (BLANK CORRECTED)

Volatile compounds	Gas bags				Fast VOST			Slow VOST		
	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Carbon tetrachloride	99.9975	99.9984	99.9964	99.9984	99.99966	99.99942	99.99946	99.9970	99.9981	99.984
Trichloroethylene	99.975	> 99.971	> 99.975	> 99.984	99.9975	99.9977	99.99906	99.985	99.991	99.9959
Benzene	a	> 99.976	> 99.88	a	a	99.972	99.914	a	99.86	99.82
Tetrachloroethylene	a	a	> 99.984	a	a	a	> 99.985	a	a	> 99.9929
Toluene	99.947	> 99.980	> 99.983	> 99.970	> 99.9973	99.9926	99.9916	> 99.966	99.941	99.957
Methyl ethyl ketone	99.9948	99.9978	99.9952	99.987	> 99.99957	99.99913	99.9979	99.9965	99.9952	99.988
Semivolatile compounds	MH5									
	Run 1	Run 2	Run 3							
Phenol	99.9985	> 99.99996	> 99.9996							
Naphthalene	99.96	99.98	99.986							
Butyl benzyl phthalate	> 99.992	> 99.995	> 99.973							
Bis (2-ethyl hexyl) phthalate	99.9985	99.993	99.996							

a Waste feed concentration < 100 µg/g in this run.

TABLE H-14. CONCENTRATIONS OF VOLATILE PICs IN STACK EFFLUENT (NOT BLANK CORRECTED), ng/L

	Run 1						
	Fast VOST ^a			Slow VOST ^a			Gas bag
	1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	
Methylene chloride	0.64	0.61	0.63	0.63	6.1	58	< 0.1
Chloroform	0.81	2.6	0.43	1.3	5.3	1.5	1.2
1,1,1-Trichloroethane	0.054	0.072	< 0.06	< 0.06	1.3	0.81	< 0.1
Chlorobenzene	0.35	2.3	0.26	0.97	1.4	0.47	0.62
							0.83
							13
	Run 2						
	Fast VOST ^a			Slow VOST ^a			Gas bag
	1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	
Methylene chloride	7.6	3.1	0.28	3.7	30	c	12
Chloroform	0.98	3.6	1.3	2.0	3.0	c	23
1,1,1-Trichloroethane	0.21	0.29	0.097	0.20	1.3	c	0.92
Chlorobenzene	1.1	2.3	0.46	1.3	0.92	c	15
							8.0
							10
	Run 3						
	Fast VOST ^a			Slow VOST ^a			Gas bag
	1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	
Methylene chloride	1.4	2.1	0.84	1.4	37	8.3	5.5
Chloroform	1.2	0.83	1.2	1.1	85	19	1.8
1,1,1-Trichloroethane	0.058	< 0.05	< 0.05	< 0.06	1.8	1.4	0.26
Chlorobenzene	0.90	0.77	0.94	0.85	2.0	2.5	1.7
							2.1
							5.6
							9.4

^a The 1st, 3rd, and 6th pairs of the fast VOST correspond to the 1st, 2nd, and 3rd pairs of the slow VOST, respectively.

^b Average of 1st and 3rd pairs.

^c Data from this sample were lost during analysis.

TABLE H-15. OUTPUT RATES FOR VOLATILE PICs IN STACK EFFLUENT (NOT BLANK CORRECTED), g/min

Run 1									
Fast VOST ^a			Slow VOST ^a						
1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	3rd pair	Avg.	Gas bag	
Methylene chloride	0.000047	0.000045	0.000046	0.000046	0.00045	0.0042	< 0.00001	< 0.0016	0.00067
Chloroform	0.000059	0.00019	0.000031	0.000095	0.00039	0.00011	0.000088	0.00020	0.000051
1,1,1-Trichloroethane	0.0000039	0.0000053	< 0.000004	< 0.000005	0.000095	0.000059	< 0.00001	< 0.00006	0.00013
Chlorobenzene	0.000026	0.00017	0.000019	0.000071	0.00010	0.000034	0.000045	0.000061	0.00092
Run 2									
Fast VOST ^a			Slow VOST ^a						
1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	3rd pair	Avg.	Gas bag	
Methylene chloride	0.00058	0.00024	0.000021	0.00028	0.0023	c	0.00091	0.0016	0.00081
Chloroform	0.000075	0.00027	0.000099	0.00015	0.00023	c	0.0017	0.00099	0.000021
1,1,1-Trichloroethane	0.000016	0.000022	0.0000074	0.000015	0.000099	c	0.000070	0.000084	0.00010
Chlorobenzene	0.000084	0.00017	0.000035	0.000099	0.000070	c	0.0011	0.00061	0.00079
Run 3									
Fast VOST ^a			Slow VOST ^a						
1st pair	3rd pair	6th pair	Avg.	1st pair	2nd pair	3rd pair	Avg.	Run 3	Run 4
Methylene chloride	0.00012	0.00018	0.000071	0.00012	0.00031	0.00070	0.0014	0.00020	0.0016
Chloroform	0.00010	0.000070	0.000010	0.000092	0.0071	0.0016	0.0030	0.000019	0.000024
1,1,1-Trichloroethane	0.0000049	< 0.000005	< 0.000005	0.000005	0.00015	0.00012	0.00022	0.00010	0.000037
Chlorobenzene	0.000076	0.000065	0.000079	0.000071	0.00017	0.00021	0.00014	0.00018	0.00079

^a The 1st, 3rd, and 6th pairs of the fast VOST corresponds to the 1st, 2nd, and 3rd pairs of the slow VOST, respectively.

^b Average of 1st and 3rd pairs.

^c Data from this sample were lost during analysis.

TABLE H-16. EMISSIONS OF SEMIVOLATILE PICs
(NOT BLANK CORRECTED)

Compound	Emission concentration (ng/L)		
	Run 1	Run 2	Run 4
<u>2,4-Dimethylphenol</u>			
Front	< 0.7	< 0.7	< 0.9
Back	< 0.7	21	< 0.9
Compound	Output rate (g/min)		
	Run 1	Run 2	Run 4
<u>2,4-Dimethylphenol</u>			
Front	< 0.00005	< 0.00005	< 0.00007
Back	< 0.00005	0.0016	< 0.00007

TABLE H-17. MODIFIED METHOD 5 TEST DATA

	Run 1, 11/3/82	Run 2, 11/4/82	Run 4, 11/5/82
Volume of gas sampled (Nm ³ , dry)	2.277	2.101	1.748
Sampling time (min)	140	140	168
Percent isokinetic	95.0	96.8	95.5
Moisture content (%)	15.8	13.0	11.9
Percent O ₂ (dry)	10.5	10.8	10.6
Percent CO ₂ (dry)	7.8	7.7	7.9
Stack flow rate (actual m ³ /min)	355	341	393
Stack temperature (°C)	809	749	810
Stack flow rate (Nm ³ /min, dry)	73	76	84
Particulate concentration			
(mg/dscm)	842	523	643
(gr/dscf)	0.367	0.228	0.280
(mg/dscm corrected to 7% O ₂)	1,125	719	865
(gr/dscf corrected to 7% O ₂)	0.491	0.313	0.378
(lb/hr)	8.1	5.2	7.2
Chloride emissions			
(g/min)	31.1	37.1	28.8
(lb/hr)	4.1	4.9	3.8

Note: Run 3 was voided by a broken probe liner plus a broken filter frit and torn filter.

Chlorides

Chloride emissions data are also presented in Table H-17. Results are expressed in output rate units based on the concentrations measured in the MM5 samples (condensate and KOH impinger). The measured chloride emissions were either slightly below or slightly in excess of the federal limitation of 4 lb/hr. However, essentially all of the chlorinated materials in the waste feed were intentionally injected into the waste for the test program and were not part of the normal waste feed at Mitchell Systems. The injection rate was set to provide sufficient POHCs for detection in the stack effluent (in the feed samples), but also with an awareness of the 4 lb/hr emission restriction.

Gaseous Effluents

Table H-18 presents a summary of the data obtained through continuous monitoring of the stack gases during the MM5 runs. Results are shown as the average and range of values for each run. In general, the four constituents monitored were relatively consistent throughout the tests. Both carbon monoxide and total hydrocarbon concentrations were very low during all three runs.

WASTE FEED ANALYSIS

Samples of both the organic and aqueous waste streams were analyzed for heating value and viscosity, as well as to determine their chloride, water and ash contents. Results are shown in Tables H-19 and H-20. An ultimate analysis was also performed on the organic waste with the results presented in Table H-21. In both cases, the data indicate that the waste streams were relatively uniform during the days of testing.

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (1) external auditing; (2) sampling QA activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade and by QA personnel. A performance audit (quantitative evaluation) and limited systems audit (qualitative evaluation) were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on:

- GC/MS analysis of volatile and/or semivolatile organics; and
- Technicon Autoanalyzer analysis of chlorides.

TABLE H-18. CONTINUOUS MONITORING DATA^a

	Run 1, 11/3/82	Run 2, 11/4/82	Run 4, 11/5/82
Oxygen (%)			
Range	7.1-11.0	8.3-11.0	7.8-12.0
Average	9.4	10.5	10.8
Carbon dioxide (%)			
Range	7.2-10.6	7.2-8.1	6.1-10.0
Average	8.5	7.6	7.4
Carbon monoxide (ppm _v)			
Range	< 1-5.8	< 1-5.3	< 1-12.9
Average	1.4	1.8	< 1
Total hydrocarbons (ppm _v) ^b			
Range	< 1	< 1	< 1-3.5
Average	< 1	< 1	< 1

^a Concentrations on dry gas basis.

^b Total HC reported as propane.

TABLE H-19. GENERAL ANALYSIS OF AQUEOUS WASTE

Parameter	Run 1	Run 2	Run 3	Run 4
Heating value, kJ/kg (Btu/lb)	1,800 780	1,720 730	1,550 660	1,590 690
% Chlorides	0.39	0.36	0.26	0.38
% Water	88.54	93.89	89.67	89.86
% Ash	0.70	0.78	0.74	0.80
Saybolt viscosity (sec)	28.9	28.2	29.5	28.7

TABLE H-20. GENERAL ANALYSIS OF ORGANIC WASTE

Parameter	Run 1	Run 2	Run 3	Run 4
Heating value, kJ/kg (Btu/lb)	34,140 14,680	34,390 14,800	37,110 15,960	34,640 14,910
% Chlorides	1.03	1.26	0.72	1.18
% Water	2.15	3.18	5.73	2.36
% Ash	1.53	2.13	2.36	0.78
Saybolt viscosity	32.1	30.1	30.3	33.1

TABLE H-21. ULTIMATE ANALYSIS OF ORGANIC WASTE

	Percent Cl	Percent C	Percent H	Percent N	Percent S	Percent Ash	Percent O (by difference)
Run 2	1.26	69.90	8.38	0.65	0.14	2.13	17.54

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics and semivolatile organics; however, the results for the quantification of bis(2-ethylhexyl)phthalate, dibenzyl butyl phthalate, phenol, and isophorone were erratic. This portion of the analytical program is judged to be within control limits, however.

Sampling QA Activities

In general, sampling QA activities are scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures included spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure used was monitoring the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX I

SUMMARY OF RESULTS FROM TESTING AT
THE DU PONT INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration which requires the collection of data on current incineration operating practices. Through a comprehensive sampling and analysis program at selected incineration facilities, test data regarding water feed composition, incinerator effluents and operating parameters are being acquired.

The EPA selected the hazardous waste incinerator at the Du Pont Pontchartrain Works in La Place, Louisiana, as one facility for the test program. The on-site sampling was conducted on November 17 and 18, 1982. Samples of the liquid waste feeds and the stack effluent were collected and analyzed. The following sections of this appendix present a summary of the emission measurement results, a brief process description, a synopsis of the test plan and the sampling and analysis methods used, and a discussion of the test program results.

SECTION 2

SUMMARY OF RESULTS

A major objective on this test program was to identify and quantify principal organic hazardous constituents (POHCs) in the waste feeds, and the POHCs and products of incomplete combustion (PICs) in the stack gas from the incinerator facility. POHCs were also quantified in ash and scrubber effluent. Particulate emissions, HCl removal efficiency, and metal emissions were also determined.

The compounds identified as POHCs and PICs were divided into two groups: volatiles and semivolatiles. POHCs in the waste feeds and stack gases were:

Volatiles

Methylene Chloride
Chloroform
1,1,1-Trichloroethane
Carbon Tetrachloride
Trichloroethylene
Tetrachloroethylene
Toluene
cis-1,4-Dichloro-2-butene

Semivolatiles

trans-1,4-Dichloro-2-butene
Benzyl chloride
Hexachloroethane
Naphthalene

PICs in the effluent gas were:

Volatiles

Benzene
Chlorobenzene
Bromodichloromethane
Dibromochloromethane
Bromoform

Semivolatiles

Phenol

The two volatile POHCs found in the highest concentration in the waste feeds were carbon tetrachloride and toluene. The volatile POHCs found in the highest concentration in the effluent were chloroform, toluene and methylene chloride. Naphthalene was the only semivolatile POHC found in the effluent. In this report, data on emission of POHCs are presented as uncorrected concentrations and emission rates accompanied by results for all "blanks", expressed in the same units.

Results of the particulate emission measurements show that the average particulate concentration (corrected to 7% O₂) for all three runs was 23 mg/dscm (0.01 gr/dscf), below the EPA regulation for hazardous waste incinerators of 180 mg/dscm (0.08 gr/dscf). Chloride removal efficiency averaged 99.9% for Runs 1 through 3, which is higher than the required removal efficiency of 99.0%. Measurable concentrations of several metals were found in the waste feed. The highest emission rates in the stack particulate were for lead and selenium.

All of these results and calculated DREs are presented in detail in Section 5.

SECTION 3

PROCESS DESCRIPTION AND PROCESS DATA

The incinerator design features both a single stage liquid injection incinerator and a rotary kiln incinerator with a natural gas fired afterburner. Each of the two incinerators has a water quench system and a cyclone which acts to remove particulate matter and droplets entrained in the exhaust gas. After exiting the cyclones the gas streams combine and pass through a common absorber system, ID fan, and stack (see Figure I-1).

A liquid organic waste is fed continuously to both the liquid injection incinerator and rotary kiln from waste feed storage tanks. The rotary kiln incinerator is also intermittently fed solid waste composed of two types of coke waste, a filter cake waste, and waste paint. These solid wastes were fed to the kiln in drums, about once every 5 min. Each drum of waste paint weighed about 10 kg while the other solid wastes ranged in weight from 70 to 160 kg. The residence time of the solids in the kiln ranged from 1 to 4 hr. Ash was removed from the kiln by a water sluice system and pumped into a clarifier. A drawing of the two combustion chambers, including thermocouple (T/C) locations, is presented in Figure I-2.

The common absorber system is a three stage scrubber system for HCl removal. Inflow water was fed into the third stage of the scrubber, the quench, and the kiln ash sluice system. Part of the effluent from the third stage was recirculated to the second stage, and the effluent from the second stage was recirculated to the first stage. The effluent from the scrubber system was combined with effluent from the quench and clarifier before being discharged to a neutralizer unit in another part of the plant.

Process data obtained during the three test runs are summarized in Table I-1. All three runs were conducted under normal operating conditions. The process data show that the liquid incinerator temperature was maintained at 1450°C for all three runs. The kiln exit temperature ranges from 750°C to 807°C, and the afterburner temperature ranges from 967°C to 1000°C. The calculated residence time of gases in the liquid incinerator averaged 0.26 sec. Gas residence time for the rotary kiln averaged 6.5 sec and for the afterburner averaged 0.72 sec. The average heat input to the liquid incinerator was 17.2×10^6 kJ/hr (16.4×10^6 Btu/hr); heat input for the rotary kiln was 18.5×10^6 kJ/hr (17.5×10^6 Btu/hr). Natural gas input to the afterburner represented a heat load of 1.2×10^6 kJ/hr (1.15×10^6 Btu/hr).

Table I-2 lists the waste feed rates for each of the five wastes used during the three runs.

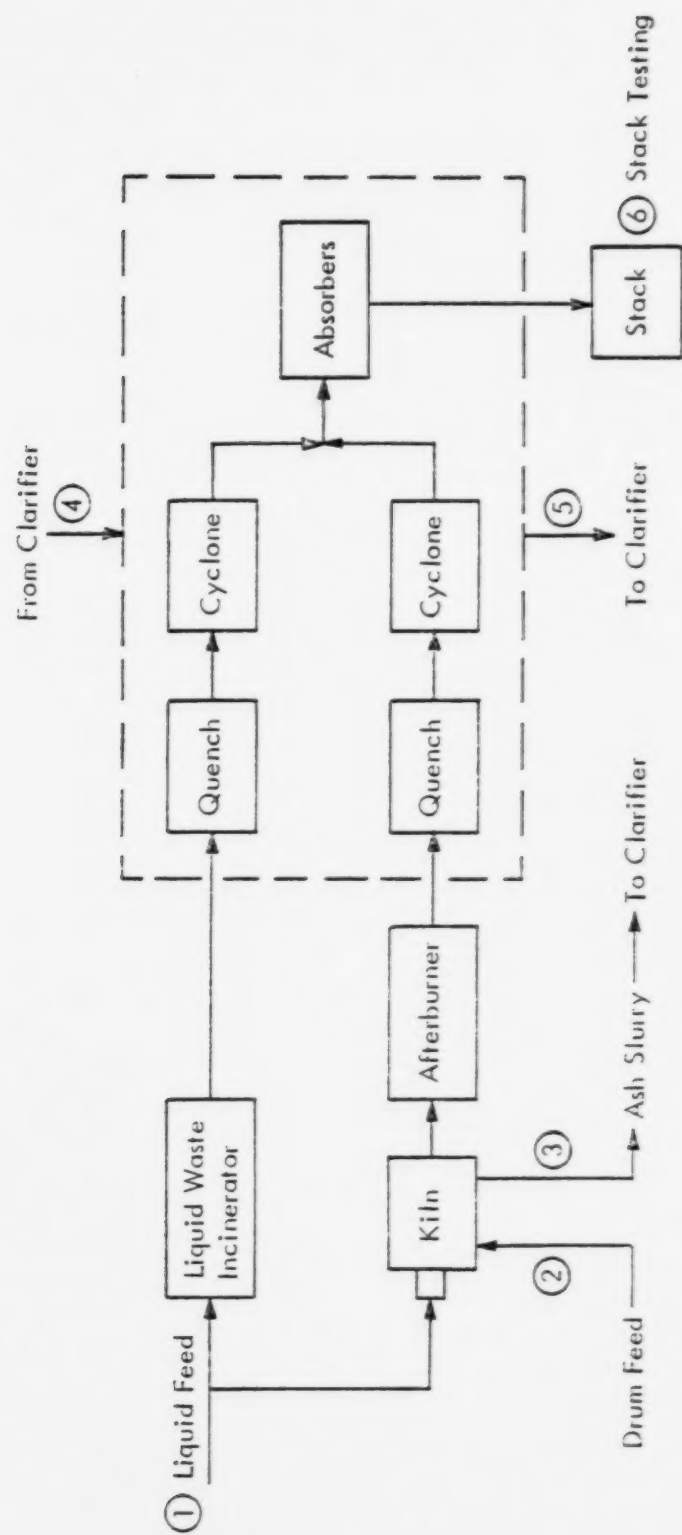
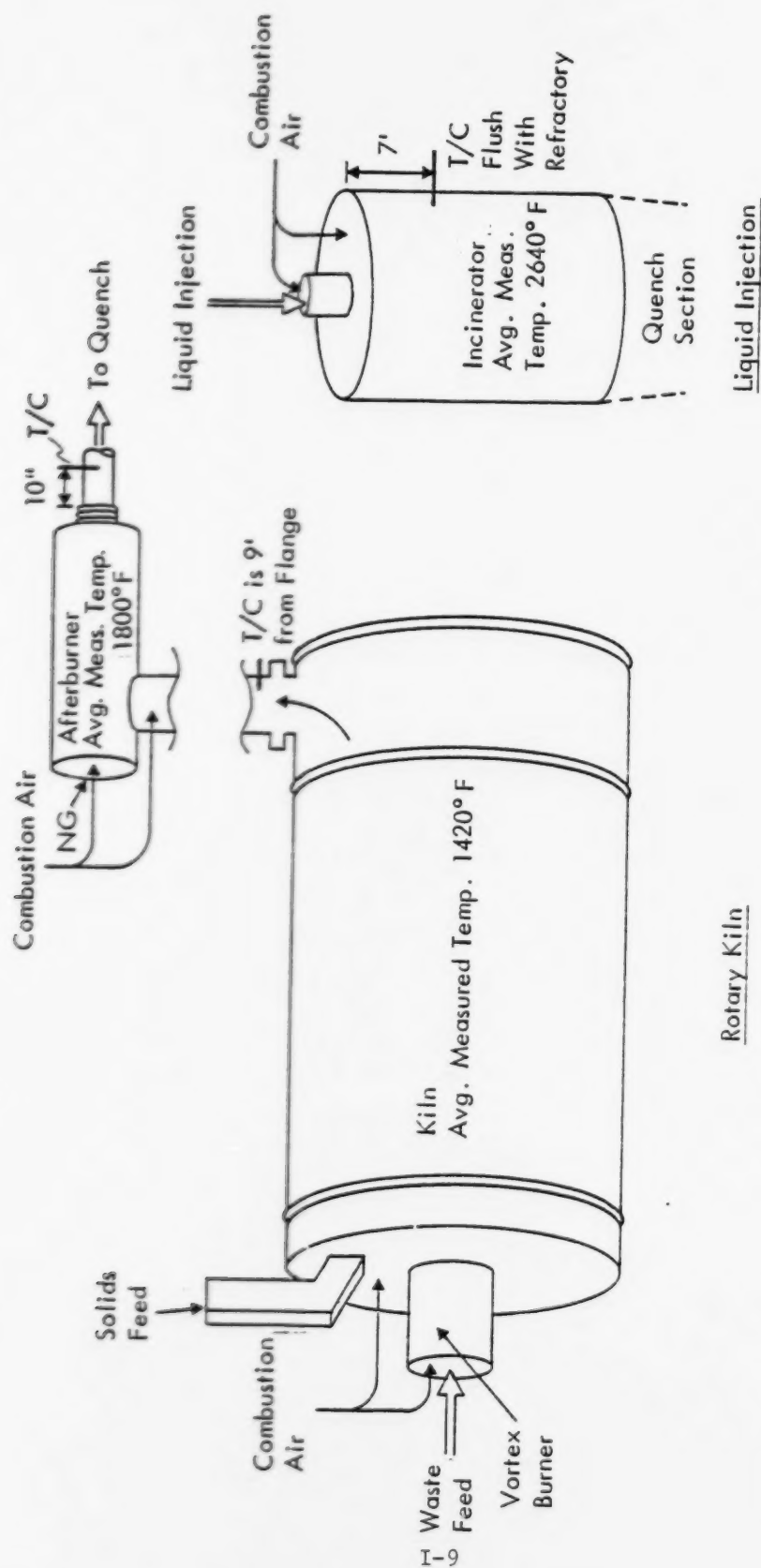


Figure I-1. Sampling points - Du Pont.



Note: T/C in kiln and afterburner extend inside, 3" past refractory
 T/C in liquid injector is flush with edge of brickwork.
 Chamber dimensions not available.

Figure I-2. Combustion chamber configurations.

TABLE I-1. PROCESS DATA

	Run 1	Run 2	Run 3
Total heat load, ^a 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	36.3 (34.4)	34.5 (32.7)	36.6 (34.7)
Kiln heat load, ^a 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	19.0 (18.0)	17.3 (16.4)	19.2 (18.2)
Liquid incinerator heat load, ^a 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	17.3 (16.4)	17.2 (16.3)	17.4 (16.5)
<u>Kiln</u>			
Atomizing airflow to kiln, m ³ /hr (SCFH)	142 (5,000)	142 (5,000)	142 (5,000)
Atomizing air pressure, kPa (PSIG)	621 (75.4)	624 (75.8)	620 (75.2)
Waste organic feed flow rate, kg/hr (lb/hr)	362 (803)	349 (773)	339 (750)
Waste organic feed pressure, kPa (PSIG)	418 (46)	418 (46)	418 (47)
Total airflow, ^b m ³ /hr (SCFH)	8,400 (296,000)	8,500 (300,000)	8,500 (300,000)
Kiln exit temperature, °C (°F)	807 (1,485)	750 (1,382)	750 (1,382)
After burner temperature, °C (°F)	1,000 (1,832)	975 (1,787)	967 (1,773)
Kiln O ₂ , %	7.1	8.0	7.2
Secondary air to kiln, m ³ /hr (SCFH)	1,400 (50,000)	1,600 (55,000)	1,600 (55,000)
Natural gas to kiln, m ³ /hr (SCFH)	0 (0)	0 (0)	0 (0)
Combustion air to kiln, m ³ /hr (SCFH)	3,500 (125,000)	3,600 (128,000)	3,500 (124,000)
Natural gas to afterburner, m ³ /hr (SCFH)	35 (1,200)	34 (1,200)	28 (1,000)
Secondary air to afterburner, m ³ /hr (SCFH)	113 (4,000)	113 (4,000)	113 (4,000)
Kiln cyclone inlet temp, °C (°F)	70 (158)	68 (154)	68 (154)
Kiln residence time, sec	6.5	6.3	6.9
Afterburner residence time, sec	0.72	0.70	0.74

(continued)

TABLE I-1. (continued)

	Run 1	Run 2	Run 3
<u>Liquid incinerator</u>			
Natural gas flow, m ³ /hr	0	0	0
(SCFH)	(0)	(0)	(0)
Atomizing air flow, m ³ /hr	176	176	176
(SCFH)	(6,200)	(6,200)	(6,200)
Atomizing air pressure, kPa	412	418	418
(PSIG)	(45)	(46)	(46)
Burner waste feed pressure, kPa	294	294	294
(PSIG)	(28)	(28)	(28)
Waste organic feed flow rate, kg/hr	542	542	542
(lb/hr)	(1,200)	(1,200)	(1,200)
Liquid burner pressure control, kPa	-1.7	-1.5	-1.7
(in of H ₂ O)	(-7)	(-6)	(-7)
Total air flow, ^b m ³ /hr	7,362	7,079	7,362
(SCFH)	(260,000)	(250,000)	(260,000)
Oxygen, %	--	--	4.4
Primary combustion air flow, m ³ /hr	6,200	6,200	6,100
(SCFH)	(220,000)	(220,000)	(215,000)
Refractory temperature, °C	1,450	1,450	1,450
(°F)	(2,642)	(2,642)	(2,642)
Incinerator residence time, sec	0.26	0.25	0.28
<u>Scrubbers</u>			
First stage scrubber pressure, kPa	-32	-32	-32
(in of H ₂ O)	(-130)	(-130)	(-130)
Stack inlet temperature, °C	70	68	70
(°F)	(158)	(154)	(158)
Exhaust blower suction pressure, kPa	-35	-35	-35
(in of H ₂ O)	(-140)	(-140)	(-140)
Third stage scrubber, water inflow, kg/hr	227	227	227
(lb/hr)	(500)	(500)	(500)
Aqueous outflow from plant, kg/hr	7,440	7,640	6,850
(lb/hr)	(16,400)	(16,800)	(15,100)

^a Based on HHV of wastes, does not include auxiliary fuel.

^b Total air flow includes air being added to the venturi scrubber.

TABLE I-2. WASTE FEED RATES, kg/min (lb/min)

	Run 1	Run 2	Run 3
Liquid waste	17.4 (38.3)	17.2 (38.0)	17.1 (37.8)
ISOM coke	0.62 (1.36)	1.19 (2.61)	1.37 (3.01)
DCB coke	0.87 (1.91)	0.53 (1.17)	1.46 (3.22)
Filter cake	2.60 (5.72)	2.09 (4.60)	1.60 (3.53)
Paint waste	1.27 (2.81)	1.23 (2.73)	1.19 (2.62)

SECTION 4

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

A description of the sampling procedures and the subsequent analyses performed on the samples is presented in Table I-3 for each sampling point shown in Figure I-1. All collected samples that were not analyzed, and the portions of samples remaining after analysis, were archived.

Organic liquid waste fed continuously to the liquid incinerator and rotary kiln was sampled every 15 min during the test runs to generate a composite sample of the waste. The solid wastes were fed to the incinerator in drums. Samples from each of the drums were taken and used to prepare a separate composite sample for each of three solid wastes (ISOM coke, DCB coke, and filter cake) during each of the three runs. A sample of each different paint waste was taken during each run and a record kept of the weight of each type fed. These data were used to prepare one composite paint waste sample for each run.

The method of sampling effluents consisted of grab sampling, except for the stack. Stack sampling was accomplished using a Modified Method 5 (MM5) train for particulates, chloride, metals, and semivolatile POHCs. Two methods were used for sampling volatile POHCs; gas bags and a volatile organic sampling train (VOST). Continuous monitors were used for O_2 , CO_2 , CO , and total hydrocarbons. A schematic diagram of the MM5 train is shown in Figure I-3, and a diagram of the VOST system is shown in Figure I-4.

Gas bag samples were collected in 15-liter Tedlar bags, drawing gas into the bags over a period of about 1 hr. Gas bags filled with prepure N_2 in the field were also prepared as gas bag blanks.

In addition to gas bags, the two VOST systems were also used to sample volatile POHCs. The VOST system consists of a Tenax trap and a backup Tenax/charcoal trap. For fast VOST, gas was drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The first pair of traps was then removed and replaced with another pair, until a total of six pairs were collected (i.e., 120 min of sampling time). Slow VOST was similar to fast VOST, but the sampling rate was lower (0.25 L/min). However, only three pair of traps were used, corresponding to the 1st, 3rd, and 6th pairs in fast VOST. Field blank sample pairs were exposed to the sampling location for the same period required to insert sample traps into the VOST apparatus. All traps were then returned to the analytical laboratory for analysis of volatile POHCs.

TABLE I-3. SUMMARY OF SAMPLING AND ANALYSIS

Sampling points	No. of samples per run	Sampling procedure	Analysis
<u>Waste Feeds</u>			
1. Liquid feed	1 composite	Collect every 15 min	HHV, ultimate ASH, viscosity POHCs Cl H ₂ O Metals
2. Drums into kiln	1 composite for each of four waste types	1 grab from each coke and sludge drum; 1 grab from every type of waste paint	As above (except viscosity)
<u>Ash</u>			
3. Kiln ash (Sluice water)	1 composite	3 grab samples	POHCs Cl Metals
<u>Scrubber and Quench Liquids</u>			
4. Influent to quench and scrubbers	1 composite	3 grabs	pH Cl POHCs and PICs
5. Effluent from quench and scrubbers	1 composite	3 grabs	As above

(continued)

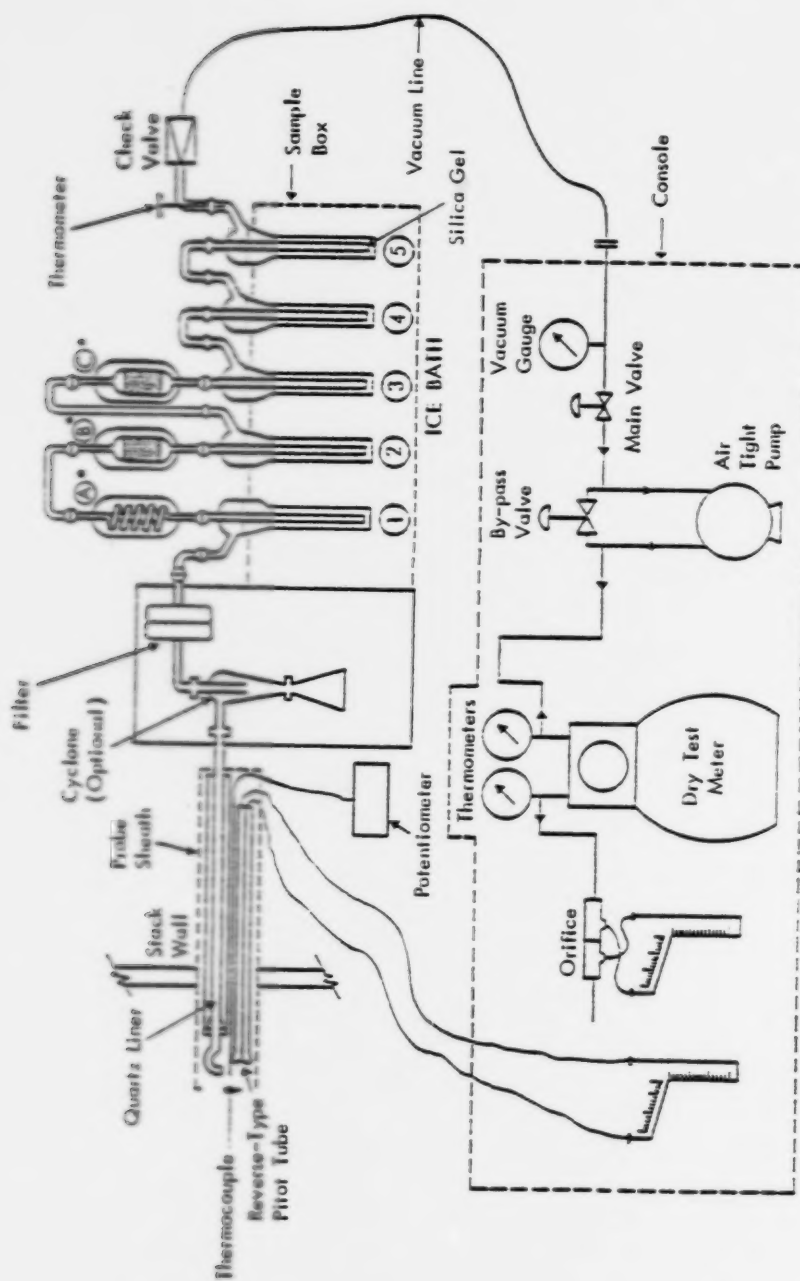
TABLE I-3. (continued)

Sampling points	No. of samples per run ^a	Sampling procedure	Analysis
<u>Stack</u>			
6. MM5 train	1	MM5 train	Particulate wt. Semivolatile POHCs PICs Cl Metals
Volatiles ^b	1	Two VOST trains (fast VOST and slow VOST)	Volatile POHCs and PICs
Volatiles	1	Gas bag	Volatile POHCs and PICs
CO ₂ and O ₂	1	Gas bag	CO ₂ and O ₂ by Orsat
CO, CO ₂ , O ₂ , and HC	Continuous	Continuous monitors	CO, O ₂ , HC

^a A total of 3 runs were carried out.

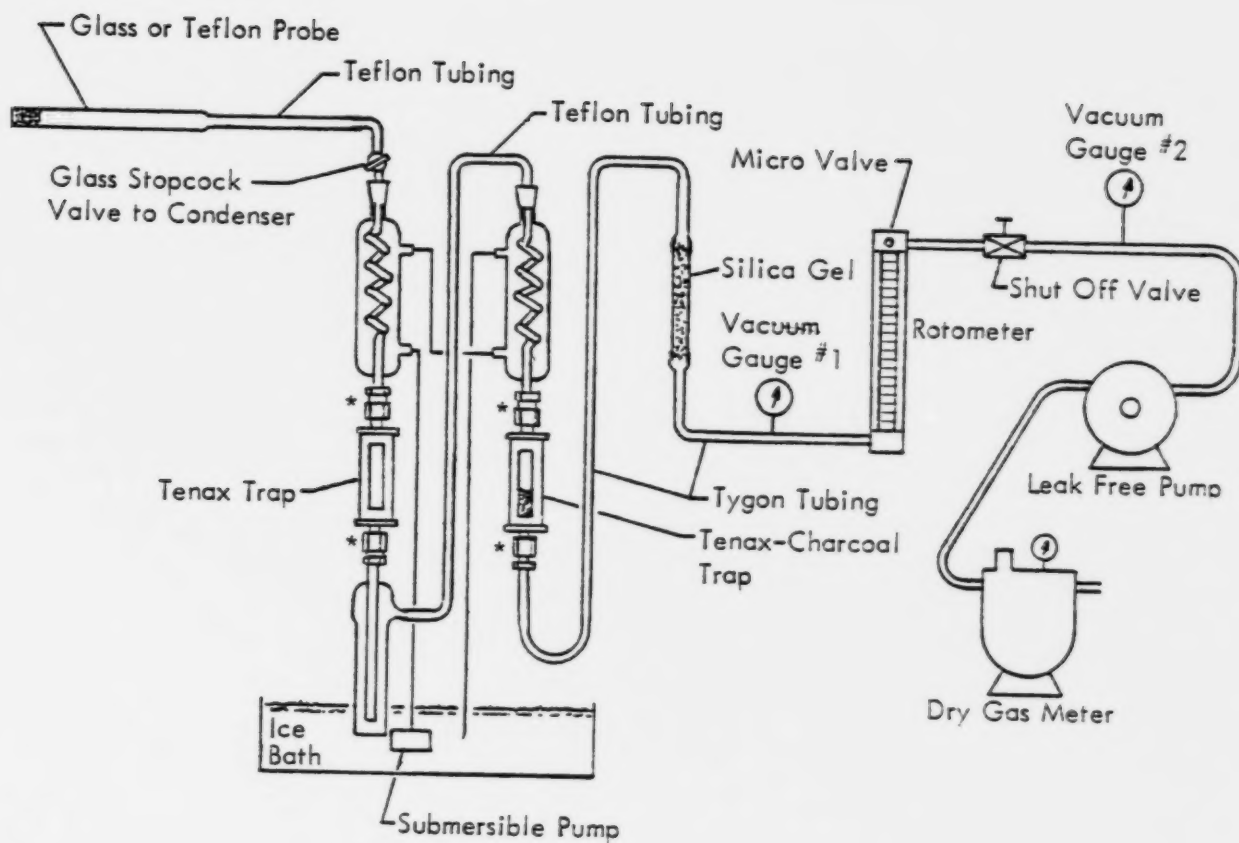
^b Two VOST trains were used during each run (fast VOST and slow VOST). Three pairs of traps were used in slow VOST, sampling 20 min at 250 ml/min with each pair. These three pairs corresponded to the 1st, 3rd, and 6th pairs used in fast VOST, sampling 20 min at 1 L/min.

VOST train samples, consisting of only one pair of traps, were also taken during one run to obtain a sample of stack ambient air and of laboratory trailer ambient air.



- ① Modified Greenburg-Smith, Reversed, Empty
 ② Greenburg-Smith, 50 ml of Double Distilled in Glass H₂O
 ③ Greenburg-Smith, 100 ml of 0.1N KOH
 ④ Modified Greenburg-Smith Empty
 ⑤ Modified Greenburg-Smith, SiO₂
 (A) Condenser
 (B) XAD Resin Cartridge #1
 (C) XAD Resin Cartridge #2
 * Ice Water Jacket

Figure I-3. Modified Method 5 sampling train (MM5).



* Special O-Ringed Stainless Steel Fittings

Figure I-4. Volatile organic sampling train (VOST).

POHCs in the waste feed and stack samples were determined by gas chromatography/mass spectrometry (GC/MS). Prior to analysis, the waste feed samples were homogenized either manually or mechanically. The homogenized feed samples were analyzed for volatile POHCs using a protocol based on Method A101b in the A. D. Little report, "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0-g aliquot of the mixed waste feed sample was dispersed in 40 mL of prepurified tetraglyme. A 10-L aliquot of the tetraglyme solution was spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 µg/g each in the original sample).

The sample was then analyzed by the purge-trap-desorb technique described in Method A101a in the A. D. Little report (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis was repeated using a larger aliquot (up to 250 µL) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs were analyzed in the waste feed samples according to Method P023 in the A. D. Little report. A 5-g portion of the homogenized waste feed sample was spiked with semivolatile surrogate compounds such as decafluorobiphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 µg/g, depending on the expected sample complexity. The sample was allowed to equilibrate with the spiked compounds and then was extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appeared on the surface of the methylene chloride, the pH was checked and the sample neutralized, if necessary. The extract was then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

An aliquot of the extract was then screened by gas chromatography (GC) with a flame ionization detector. If necessary, the extract was further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column. A 1.0-mL aliquot of the extract was then spiked with 20 µg of D₁₀-anthracene internal standard and analyzed by fused silica capillary column GC/MS.

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with VOST. A known amount of the gas bag sample, usually 5.0 L, was transferred onto a Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). To make this transfer, a needle was inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were then analyzed using the same techniques that are described below for the VOST traps.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal absorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene internal standards using the flash vaporization technique, in which the spiking solution is vaporized and carried onto the trap with a carrier gas. In order to accomplish this spiking, the glass traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min,

was turned on through the trap and a syringe needle injected through the GC septum port. Slowly, 1 μ L of a 25-ng/ μ L methanolic solution of the internal standards was expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was only about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL), which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds passed into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs in the stack involved several components of the MM5 train. Each of the components of the MM5 train was spiked with a separate surrogate standard according to the following scheme:

Front XAD-2 resin trap - 20 μ g of 13C-tetrachlorobiphenyl
Back XAD-2 resin trap - 20 μ g of decafluorobiphenyl
Particulate filter plus probe rinse residue - 20 μ g of D₁₂-chrysene
Condensate water - 20 μ g of D₈-naphthalene

The spiked components were normally extracted separately and the extracts combined, dried with anhydrous sodium sulfate, concentrated to 1.0 mL using Kuderna-Danish evaporation according to Method P031 in the A. D. Little report and using supplementary gentle nitrogen blowdown, and analyzed by fused silica capillary column GC/MS. The samples were extracted according to the following procedures:

XAD-2 resin traps: The XAD-2 resin traps were Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Particulate filters plus probe rinse residues: The residue from the acetone rinse of the probe was added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Condensate water: The condensate water was extracted with three 60-mL aliquots of methylene chloride as described in Method P021a in the A. D. Little report (equivalent to EPA Method 625).

POHCs in scrubber water and ash samples were analyzed using the procedures described above. To analyze for metals, the samples were acid digested and analyzed by inductively coupled argon plasma emission spectrophotometry and/or atomic absorption spectrophotometry. Particulate and

chloride analyses were conducted according to EPA Method 5 and EPA Method 325.2, respectively, from "Methods for Chemical Analysis of Water and Wastes." EP toxicity samples were tested according to Federal Register, 45, May 19, 1980. Water content in the waste feeds was determined by the Karl Fischer titration method. Chlorine in the waste feed and fabric filter ash was determined by potentiometric titration using a Fisher Titralizer Model 41. Heating value was determined by ASTM Methods D240-64 and D-2015-66. Orsat analyses were performed according to EPA Method 3. Continuous monitoring involved the following:

- O₂ - Beckman Model 742 (polarographic sensor)
- CO - Beckman Model 215A (nondispersive infrared)
- HC - Beckman Model 402 (heated oven flame ionization detector)
- CO₂ - Horiba Model PIR-2000S (nondispersive infrared)

In Appendix B, descriptions of all the above sampling and analysis methods are presented in more detail.

SECTION 5

DISCUSSION OF RESULTS

INPUT AND OUTPUT CONCENTRATIONS AND RATES FOR POHCs

POHC input and output concentrations and mass rates were determined for all POHCs listed by EPA which were detected in the waste feeds at concentrations above 100 µg/g. No attempt was made to restrict the number of POHCs based on any selection or ranking method. All POHCs reported are compounds that were quantified against authentic standards. In all tables, values marked < indicate detection levels.

The concentrations of volatile and semivolatile POHCs in the waste feeds are given in Tables I-4 and I-5. Input rates for the POHCs are presented in Table I-6, based upon the POHC concentration values in Tables I-4 and I-5, and the waste feed rates presented in Table I-2.

Tables I-7 and I-8 present volatile POHC output data (not blank corrected) for gas bags, slow VOST and fast VOST samples. Table I-7 lists the output concentrations in ng/L, and Table I-8 shows the output rates in g/min. The concentrations of a few of the POHCs and PICs in the effluent could be contributed to by the stripping of some volatiles from the scrubber water. This will be discussed in a later section. Table I-9 presents corresponding values for semivolatile output concentrations and output rates, also not blank corrected.

Destruction and removal efficiencies (DREs), all POHCs based on the above data, are presented in Table I-10. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$DRE = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

EMISSION OF PICs

Analysis of samples included identification of PICs, i.e., those compounds identified in the stack samples that are listed as POHCs but which were not found in the waste feed samples at concentrations above 100 µg/g. The output concentration and rate for volatile PICs are tabulated in Tables I-11 and I-12. The output concentration and rate data for the semivolatile PIC are shown in Table I-13. Benzene exhibited the highest concentrations and emission rates of the PICs.

TABLE 1-4. CONCENTRATIONS OF VOLATILE POCs IN WASTE FEEDS ($\mu\text{g/g}$)

Compound	Run No.	Liquid waste			Filter cake			DCB coke			ISON coke			Paint waste		
		1	2 ^a	3	1	2	3	1	2	3	1	2	3	1	2	3
Methylene chloride	21,000	21,000	25,000	9,200	17	100	3,400	290	60	1,900	150	35	450	470	260	
Chloroform	4,100	2,900	5,400	950	3.6	5.1	120	28	3.2	100	< 10	< 10	460	670	12	
1,1,1-Trichloro-ethane	< 100	< 100	< 100	< 10	< 10	19	250	< 10	5.4	< 10	< 10	< 10	< 20	< 20	< 20	
Carbon tetra-chloride	81,000	68,000	72,000	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 20	< 40	< 40	< 40	
Trichloroethylene	3,600	4,000	2,600	160	40	14	< 10	< 10	< 10	< 10	< 10	< 10	< 20	< 20	< 20	
Tetrachloroethylene	14,000	11,000	11,000	220	15	22	58	68	2.0	160	4.5	7.4	< 20	< 20	63	
Toluene	270,000	240,000	270,000	16,000	20,000	6,700	< 10	< 10	160	< 10	1,400	620	130,000	280,000	300,000	
cis-1,4-Dichloro-2-butene	21,000	18,000	23,000	740	45	84	630	750	3,200	290	430	3,900	< 40	< 40	< 40	

^a Average of three replicates.

TABLE 1-5. CONCENTRATION OF SEMIVOLATILE PHCHs IN WASTE FEEDS (µg/g)

Compound	Run No.	Liquid waste			Filter cake			DCB coke			ISOM coke			Paint waste		
		1	2	3	1	2	3	1	2	3	1	2	3	1	2	3
trans-1,4-Dichloro-2-butene	59,000	59,000	70,000	2,400	3,400	130	8,300	8,800	11,000	14,000	3,300	9,500	< 60	< 60	< 60	< 60
Benzyl chloride	2,700	3,000	2,900	200	230	330	< 30	< 30	< 30	140	< 60	< 60	< 30	< 30	< 30	< 30
Hexachloroethane	590	560	500	< 90	< 90	< 90	< 200	< 90	< 90	< 200	< 90	< 200	< 90	< 90	< 90	< 30
Naphthalene	< 100	< 100	< 100	960	910	820	36	40	< 30	< 60	47	< 60	< 30	47	< 30	< 30

TABLE I-6. CALCULATED INPUT RATE FOR POHCs IN WASTE FEEDS
(g/min)

	Run 1	Run 2	Run 3
<u>Volatile POHCs</u>			
Methylene chloride	390	360	430
Chloroform	75	51	92
1,1,1-Trichloroethane ^a	0.22	NA	NA
Carbon tetrachloride	1,400	1,200	1,200
Trichloroethylene	63	69	45
Tetrachloroethylene	240	190	190
Toluene	4,900	4,500	5,000
<u>cis-1,4-Dichloro-2-butene</u>	370	310	400
<u>Semivolatile POHCs</u>			
<u>trans-1,4-Dichloro-2-butene</u>	1,000	1,000	1,200
Benzyl chloride	48	52	50
Hexachloroethane	10	10	9
Naphthalene	2.5	2.0	1.3

^a 1,1,1-Trichloroethane was detected at a concentration greater than 100 µg/g only in one waste stream in Run 1.

TABLE I-7. CONCENTRATION OF VOLATILE POHCs IN STACK EFFLUENT (NOT BLANK CORRECTED), ng/L

Run 1									
	Gas bag	Fast VOST			Average	Slow VOST			Average
		1st pair ^b	3rd pair ^c	6th pair ^d		1st pair ^b	2nd pair ^c	3rd pair ^d	
Methylene chloride	6.4	9.5	3.4	14	9.0	12	2.1	6.3	6.8
Chloroform	22	17	13	30	20	21	0.65	17	13
1,1,1-Trichloroethane	< 0.2	0.70	0.24	0.34	0.43	0.65	0.42	< 0.4	< 0.49
Carbon tetrachloride	9.8	4.2	2.9	4.5	3.9	6.9	4.8	4.1	5.3
Trichloroethylene	14	IF	IF	0.51	-	1F	2.9	2.6	-
Tetrachloroethylene	5.8	3.2	2.7	6.6	4.2	1.2	8.3	1.1	3.5
Toluene	36	30	28	39	32	11	41	7.9	20
cis-1,4-Dichloro-2-butene ^a	< 0.2	5.7	2.0	1.1	2.9	0.6	2.1	< 0.4	< 1

Run 2										
	Gas bag	Fast VOST			Average	Slow VOST			Average	Lab trailer air (fast VOST, 1 pair)
		1st pair ^b	3rd pair ^c	6th pair ^d		1st pair ^b	2nd pair ^c	3rd pair ^d		
Methylene chloride	3.4	1.9	5.5	7.3	4.9	4.9	12	14	10	9.1
Chloroform	19	14	16	16	16	17	20	19	19	0.80
1,1,1-Trichloroethane	0.94	0.30	0.10	0.20	0.20	< 0.4	< 0.4	0.3	< 0.4	2.4
Carbon tetrachloride	IF	3.0	2.5	1.5	2.3	4.5	4.4	3.1	4.0	0.70
Trichloroethylene	26	0.59	0.36	1F	0.48	2.4	1.3	2.0	1.9	2.7
Tetrachloroethylene	4.0	2.2	1.4	2.6	2.1	1.1	1.5	2.0	1.5	2.7
Toluene	9.6	7.5	5.4	14	9.0	4.3	8.2	17	9.8	13
cis-1,4-Dichloro-2-butene ^a	< 0.2	0.09	0.08	< 0.09	< 0.08	< 0.4	< 0.4	0.4	< 0.4	2.3

(continued)

TABLE I-7. (continued)

Run 3

	Gas bag	Fast VOST				Slow VOST				Stack amb. air (slow VOST, 1 pair)
		1st pair ^b	3rd pair ^c	6th pair ^d	Average	1st pair ^b	2nd pair ^c	3rd pair ^d	Average	
Methylene chloride	16	6.7	11	23	14	9.2	22	19	17	0.76
Chloroform	22	17	16	30	20	21	24	22	22	3.4
1,1,1-Trichloroethane	< 0.2	0.49	0.19	0.26	0.31	< 0.4	< 0.5	< 0.6	< 0.5	3.4
Carbon tetrachloride	17	6.4	6.7	12	8.4	8.5	8.1	3.0	6.5	1.8
Trichloroethylene	15	2.8	1.6	3.1	2.5	11	3.6	4.0	6.2	2.2
Tetrachloroethylene	28	3.3	3.1	6.3	4.2	4.4	4.2	3.8	4.1	3.0
Toluene	28	14	14	15	14	24	22	15	20	30
cis-1,4-Dichloro-2-butene ^a	< 0.20	0.18	< 0.09	< 0.09	< 0.12	< 0.5	< 0.4	< 0.4	< 0.5	0.4

Note: "Less than" values can vary due to the mathematics of adding a quantified value on one trap of a pair to a greater than value on the other trap of the pair. They also are due to the different amounts of gas sampled by fast VOST (20 L), slow VOST (5 L) and bags (5 L).

^a trans-1,4-Dichloro-2-butene has also been quantitated, as a semivolatile POC (see Table 14).

^b Fast VOST 1st pair corresponds to slow VOST 1st pair.

^c Fast VOST 3rd pair corresponds to slow VOST 2nd pair.

^d Fast VOST 6th pair corresponds to slow VOST 3rd pair.

TABLE 1-8. VOLATILE FORC EMISSION RATES (NOT BLANK CORRECTED), g/min

Gas bag	Run 1				
	Fast VOST			Slow VOST	
	1st pair	3rd pair	6th pair	Average	1st pair 2nd pair 3rd pair Average
Methylene chloride	0.0024	0.0035	0.0013	0.0052	0.0044 0.0077 0.0023 0.0025
Chloroform	0.0081	0.0063	0.0048	0.011	0.0074 0.0077 0.0024 0.0048
1,1,1-Trichloroethane	< 0.000074	0.00026	0.000089	0.00013	0.00016 0.00024 0.00015 < 0.00018
Carbon tetrachloride	0.0036	0.0015	0.0011	0.0017	0.0014 0.0025 0.0018 0.0020
Trichloroethylene	0.0052	IF	IF	0.00019	IF 0.0011 0.00096 -
Tetrachloroethylene	0.0021	0.0012	0.0010	0.0024	0.0015 0.00044 0.0031 0.0013
Toluene	0.013	0.011	0.010	0.014	0.012 0.0041 0.015 0.0074
cis-1,4-Dichloro-2-butene ^a	< 0.000074	0.0021	0.00074	0.00041	0.0011 0.00022 0.00077 < 0.00015 < 0.000369

Gas bag	Run 2				
	Fast VOST			Slow VOST	
	1st pair	3rd pair	6th pair	Average	1st pair 2nd pair 3rd pair Average
Methylene chloride	0.0013	0.00071	0.0021	0.0027	0.0018 0.0018 0.0052 0.0037
Chloroform	0.0071	0.0052	0.0060	0.0060	0.0064 0.0075 0.0071 0.0071
1,1,1-Trichloroethane	0.00035	0.00011	0.000037	0.000075	< 0.00015 < 0.00015 < 0.00015
Carbon tetrachloride	IF	0.0011	0.00094	0.00056	0.0017 0.0016 0.0012 0.0015
Trichloroethylene	0.0097	0.00022	0.00013	IF	0.00090 0.00049 0.00075 0.00071
Tetrachloroethylene	0.0015	0.00082	0.00053	0.00097	0.00041 0.00056 0.00075 0.00056
Toluene	0.0036	0.0028	0.0020	0.0052	0.0016 0.0031 0.0064 0.0037
cis-1,4-Dichloro-2-butene ^a	< 0.000075	0.000013	0.000010	< 0.000014	< 0.00015 < 0.00015 < 0.0015

(continued)

TABLE 1-8. (continued)

	Gas bag	Fast VOST			Run 3			Slow VOST		
		1st pair	3rd pair	6th pair	Average	1st pair	2nd pair	3rd pair	Average	
Methylene chloride	0.0057	0.0024	0.0040	0.0082	0.0050	0.0033	0.0078	0.0068	0.0061	
Chloroform	0.0078	0.0061	0.0057	0.011	0.0076	0.0075	0.0085	0.0078	0.0078	
1,1,1-Trichloroethane	< 0.000071	0.00017	0.000068	0.000093	0.00011	< 0.00014	< 0.00018	< 0.00021	< 0.00018	
Carbon tetrachloride	0.0061	0.0023	0.0024	0.0043	0.0030	0.0031	0.0029	0.0011	0.0023	
Trichloroethylene	0.0053	0.0010	0.00057	0.0011	0.00090	0.0040	0.0013	0.0014	0.0022	
Tetrachloroethylene	0.010	0.0011	0.0011	0.0022	0.0015	0.0016	0.0015	0.0014	0.0015	
Toluene	0.010	0.0050	0.0050	0.0053	0.0050	0.0085	0.0078	0.0053	0.0071	
cis-1,4-Dichloro-2-butene ^a	< 0.000071	0.000064	< 0.000032	< 0.000012	< 0.000043	< 0.00018	< 0.00014	< 0.00014	< 0.00018	

^a Trans-1,4-Dichloro-2-butene has also been quantitated, as a semivolatile POC.

TABLE I-9. EMISSIONS OF SEMIVOLATILE POHCs
(NOT BLANK CORRECTED)

Compound		Emission concentration (ng/L)		
		Run 1	Run 2	Run 3
<u>trans</u> -1,4-Dichloro-2-butene	Front half	< 2	< 2	< 2
	Back half	< 0.9	< 0.9	< 0.9
Benzyl chloride	Front half	< 0.9	< 0.9	< 0.9
	Back half	< 0.4	< 0.4	< 0.5
Hexachloroethane	Front half	< 3	< 3	< 3
	Back half	< 1	< 1	< 1
Naphthalene	Front half	< 0.9	< 0.9	< 0.9
	Back half	150	61	110
		Output rate (g/min)		
<u>trans</u> -1,4-Dichloro-2-butene	Front half	0.0007	< 0.0007	< 0.0007
	Back half	< 0.0003	< 0.0003	< 0.0003
	Total	< 0.001	< 0.001	< 0.001
Benzyl chloride	Front half	< 0.0003	< 0.0003	< 0.0003
	Back half	< 0.0001	< 0.0001	< 0.0002
	Total	< 0.0004	< 0.0004	< 0.0005
Hexachloroethane	Front half	< 0.001	< 0.001	< 0.001
	Back half	< 0.0004	< 0.0004	< 0.0004
	Total	< 0.001	< 0.001	< 0.001
Naphthalene	Front half	< 0.0003	< 0.0003	< 0.0003
	Back half	0.055	0.023	0.039
	Total	0.055	0.023	0.039

TABLE 1-10. DESTRUCTION AND REMOVAL EFFICIENCIES (PERCENT) FOR THE HOT POINT INCINERATION (BLANK CORRECTED)

Volatile compounds	Gas bags			Fast WWT			Slow WWT		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
Methylene chloride	> 99.99919	> 99.99965	> 99.99987	99.99919	99.99956	99.9989	> 99.99941	> 99.9991	> 99.9988
Chloroform	99.989	99.986	99.9915	99.9929	99.989	99.9917	> 99.9938	99.987	99.9914
1,1,1-Trichloroethane	> 99.966	a	a	99.928	a	a	> 99.932	a	a
Carbon tetrachloride	99.99979	IF	99.99956	99.99990	99.99928	99.99976	99.99986	99.99988	99.99981
Trichloroethylene	> 99.9917	99.9907	> 99.988	99.99971	99.99975	99.9985	99.9984	99.9990	99.9951
Tetrachloroethylene	> 99.99911	> 99.99922	99.9951	99.99937	99.99960	99.99921	> 99.99948	> 99.99972	99.99926
Toluene	99.99980	> 99.99921	> 99.99980	99.99975	99.99926	99.99902	99.99986	> 99.99926	99.99986
cis-1,4-Dichloro-2-butene	> 99.99994	> 99.99994	> 99.99994	99.99971	> 99.99991	> 99.99991	> 99.99990	> 99.99998	> 99.99998
Semi-volatile compounds									
	Run 1	Run 2	Run 3						
Trans-1,4-Dichloro-2-butene	> 99.99990	> 99.99990	> 99.99992						
Benzyl chloride	> 99.99996	> 99.99996	> 99.99994						
Hexachloroethane	> 99.99	> 99.99	> 99.99						
Naphthalene	98.0	99.10	97.4						

IF = Quantitation prohibited due to interfering compound on GC/MS.

a Waste feed concentration < 100 µg/g in this run.

TABLE 1-11. CONCENTRATION OF VOLATILE PICs IN STACK EFFLUENT
(NOT BLANK CORRECTED), ng/l.

		Run 1						
Gas bag		Fast VOST			Slow VOST			Average
		1st pair	3rd pair	6th pair	1st pair	2nd pair	3rd pair	
Benzene	310	2,200 ^a	2,600 ^a	52	1,600	1,200 ^a	1,800 ^a	370 ^a
Chlorobenzene	11	10	9.5	10	9.8	2.7	9.0	2.3
Bromodichloro- methane	5.6	3.7	3.0	6.2	4.3	1.5	4.8	1.8
Dibromochloro- methane	1.4	1.1	0.9	< 0.1	< 0.4	< 0.4	1.3	< 0.4
Bromoform	< 0.2	0.22	0.06	0.07	0.12	< 0.4	< 0.4	< 0.4
Run 2								
Gas bag		Fast VOST			Slow VOST			Average
		1st pair	3rd pair	6th pair	1st pair	2nd pair	3rd pair	
Benzene	98 ^a	9.3	7.5	780 ^a	11	13	240 ^a	90
Chlorobenzene	2.0	0.80	0.43	4.4	0.61	0.94	7.1	2.9
Bromodichloro- methane	2.6	1.2	2.4	2.7	0.7	1.0	1.0	0.9
Dibromochloro- methane	0.8	1.0	1.0	0.8	< 0.6	1.1	0.9	< 0.9
Bromoform	< 0.2	0.12	0.10	0.09	< 0.4	< 0.4	< 0.4	< 0.4

(continued)

TABLE 1-11. (continued)

Run 3									
Gas bag	Fast VOST			Average	1st pair	2nd pair	Slow VOST		Stack amb. air (slow VOST, 1 pair)
	1st pair	3rd pair	6th pair				2nd pair	3rd pair	
Benzene	400 ^a	110 ^a	200 ^a	130	130 ^a	150	190 ^a	160	20
Chlorobenzene	5.8	3.8	4.2	3.9	0.4	4.8	4.8	3.3	1.9
Bromodichloro- methane	3.0	2.3	3.9	2.9	2.3	3.4	2.4	2.7	< 0.4
Dibromochloro- methane	2.6	1.3	1.8	1.4	1.2	0.6	0.8	0.9	< 0.4
Bromoform	0.40	0.66	0.29	0.41	< 0.5	< 0.4	< 0.4	< 0.4	< 0.4

^a The quantification of these numbers was based on a minor ion in the GC/MS, and therefore they are not as precise as the other numbers presented.

TABLE I-12. VOLATILE PIC EMISSION RATES (NOT BLANK CORRECTED), g/min

Gas bag	Run 1					Run 2					Run 3				
	Fast Vial					Fast Vial					Fast Vial				
	1st pair	2nd pair	3rd pair	Average	1st pair	1st pair	2nd pair	3rd pair	Average	1st pair	1st pair	2nd pair	3rd pair	Average	1st pair
Benzene	0.12	0.81 ^a	0.96 ^a	0.019	0.019	0.019	0.019	0.019	0.019	0.019	0.019	0.019	0.019	0.019	0.019
Chlorobenzene	0.0041	0.0017	0.0035	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037	0.0037
Bromodichloromethane	0.0021	0.0014	0.0011	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023	0.0023
Dibromochloromethane	0.00052	0.00041	0.00031	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037	< 0.00037
Bromoform	< 0.000074	0.00081	0.00022	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026	0.000026
Benzene	0.017 ^a	0.0015	0.0028	0.016	0.016	0.016	0.016	0.016	0.016	0.016	0.016	0.016	0.016	0.016	0.016
Chlorobenzene	0.00075	0.00030	0.00016	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010
Bromodichloromethane	0.00097	0.00065	0.00040	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037	0.00037
Dibromochloromethane	0.00030	0.00017	0.00009	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010
Bromoform	< 0.000075	0.000065	0.000037	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036	0.000036
Benzene	0.14 ^a	0.027 ^a	0.039 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a	0.071 ^a
Chlorobenzene	0.0021	0.0014	0.0014	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015	0.0015
Bromodichloromethane	0.0011	0.00082	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085	0.00085
Dibromochloromethane	0.00093	0.00046	0.00036	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046	0.00046
Bromoform	0.00014	0.00023	0.00011	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010	0.00010

^a The quantification of these numbers was based on a minor ion in the GC/MS, and therefore they are not as precise as the other numbers presented.

TABLE I-13. EMISSIONS OF SEMIVOLATILE PICs
(NOT BLANK CORRECTED)

Compound		Emission concentration (ng/L)		
		Run 1	Run 2	Run 3
Phenol	Front half	< 0.9	< 0.9	< 0.9
	Back half	22	18	27
		Output rate (g/min)		
Phenol	Front half	< 0.0003	< 0.0003	< 0.0003
	Back half	0.0081	0.0067	0.0096
	Total	0.0081	0.0067	0.0096

BLANK CORRECTION VALUES FOR POHCs AND PICs

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table I-14. The sample volumes for gas bags as well as slow and fast VOST are presented in Table I-15. Blank correction data for semivolatile POHCs and PICs are presented in Table I-16. Sample volumes for the MM5 train can be found in Table I-17.

PARTICULATE EMISSIONS

Particulate emissions were measured using a Modified Method 5 train. The data associated with the MM5 train for the three runs are presented in Table I-17. Particulate emission data are presented in Table I-18. The average outlet particulate concentration during the three runs was 23 mg/dscm (corrected to 7% O₂), compared to 180 mg/dscm in EPA's regulation. The total particulate emission rate averaged 0.39 kg/hr over the three runs.

CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

Table I-19 summarizes chloride input and output rates. The data show an average removal efficiency of 99.9% for the three runs. This compares favorably with the EPA regulation of 99.0% chloride removal efficiency. The output rate was never greater than 0.407 kg/hr for the three runs, well below the EPA maximum output rate of 1.8 kg/hr.

CONTINUOUS MONITORING DATA

Continuous monitors were used to determine concentration of O₂, CO₂, CO, and HC in the stack effluent throughout the test periods. The results are summarized in Table I-20. The concentrations of CO and HC were relatively high throughout the three runs, and it was observed that they increased whenever a drum of paint waste was fed to the rotary kiln.

CHLORIDES IN SCRUBBER WATER STREAMS

The tests included sampling of the scrubber water inflow and outlet streams, as well as the kiln ash sluice water. The chloride concentrations in these three streams are presented in Table I-21. The pH of the outlet water was measured at 1 in all runs.

POHCs AND PICs IN SCRUBBER WATER STREAMS

The scrubber water streams were also analyzed for the presence of volatile and semivolatile POHCs. The results are presented in Table I-22. Four volatiles--methylene chloride, chloroform, bromodichloromethane, and dibromochloromethane--appear in greater concentrations in the inflow stream. These data suggest that they are being stripped from the inflow waters by the effluent gas. Assuming that the inflow rate was the same as the outflow from the scrubber, quench, and kiln ash sluice systems, the concentration difference between the inflow and outflow water streams could account for all of the chloroform detected in the stack.

TABLE I-14. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>				
Methylene chloride	320	550	10	8.7
Chloroform	5.5	7.8	2.1	1.6
1,1,1-Trichloroethane	2.2	3.8	0.23	0.81
Carbon tetrachloride	10	6.5	< 1	0
Trichloroethylene	44	26	0.18	0.53
Tetrachloroethylene	8.8	7.2	0.77	2.3
Toluene	47	36	6.4	6.0
cis-1,4-Dichloro-2-butene	< 1	0	< 1	0
<u>PICs</u>				
Benzene	28	39	11	12
Chlorobenzene	2.4	1.5	2.3	1.7
Bromodichloromethane	< 1	0	< 1	0
Dibromochloromethane	< 1	0	< 1	0
Bromoform	< 1	0	< 1	0

TABLE I-15. VOLATILE SAMPLE VOLUMES
(DRY STANDARD LITERS)

Gas bags		Run No.	VOST			
Run No.	Volume (L)		Fast		Slow	
			Pair No.	Volume (L)	Pair No.	Volume (L)
1	5.0	1	1	21.7	1	4.9
2	5.0	1	3	21.1	2	5.2
3	5.0	1	6	21.1	3	5.1
		2	1	21.1	1	5.1
		2	3	21.2	2	5.0
		2	6	21.9	3	4.9
		3	1	20.3	1	4.4
		3	3	21.3	2	5.0
		3	6	20.5	3	5.0

TABLE I-16. SEMIVOLATILE BLANK CORRECTION VALUES

Compound	Blank correction value (µg)
<u>POHCs</u>	
trans-1,4-Dichloro-2-butene	< 2
Benzyl chloride	< 1
Hexachloroethane	< 3
Naphthalene	34
<u>PICs</u>	
Phenol	< 2

TABLE I-17. METHOD 5 TEST DATA

	Run 1	Run 2	Run 3
Vol. of gas sampled, Nm ³ (dscf)	2.278 (80.44)	2.296 (81.08)	2.196 (77.55)
Sampling time (min)	160	160	160
Percent isokinetic	100.5	99.8	100.5
Moisture content (%)	4.4	4.4	5.1
Percent O ₂ (dry)	9.8	10.6	10.0
Percent CO ₂ (dry)	8.6	8.0	8.6
Stack temperature, °C (°F)	69 (157)	67 (153)	70 (157)
Stack flow rate, act. m ³ /min (act. ft ³ /min)	451 (15,900)	456 (16,100)	438 (15,500)
Stack flow rate, Nm ³ /min (dscf/min)	369 (13,000)	374 (13,200)	356 (12,600)
Stack velocity, m/min (ft/min)	243 (798)	246 (807)	236 (774)
Stack height, m (ft)	34 (110)	34 (110)	34 (110)

TABLE I-18. PARTICULATE CONCENTRATION AND EMISSION RATE DATA

	Particulate concentration		
	Run 1	Run 2	Run 3
<u>Measured</u>			
mg/dscm	26.9	7.7	19.5
(gr/dscf)	(0.0117)	(0.0034)	(0.0085)
<u>Corrected^a</u>			
mg/dscm	33.7	10.4	24.8
(gr/dscf)	(0.0147)	(0.0045)	(0.0108)
	Particulate emissions		
	Run 1	Run 2	Run 3
<u>Emission rate</u>			
kg/hr	0.59	0.17	0.42
(lb/hr)	(1.31)	(0.38)	(0.91)

^a Measured emissions were corrected to 7% O₂ as follows:

$$C_{\text{corr}} = \frac{(14\%)}{(21\% - \%O_2)} (C_{\text{measured}})$$

TABLE I-19. CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

	Run 1	Run 2	Run 3
<u>Chloride emissions</u>			
kg/hr	0.235	0.295	0.407
(lb/hr)	(0.518)	(0.651)	(0.896)
<u>Chloride in feed</u>			
kg/hr	287	290	305
(lb/hr)	(633)	(639)	(673)
<u>Removal efficiency</u>			
(Based on chloride in feed)	99.92%	99.90%	99.87%

TABLE I-20. CONTINUOUS MONITORING DATA^a

	Run 1	Run 2	Run 3
Oxygen (%)			
Range	7.0-10.6	7.4-10.9	7.3-12.7
Average	9.2	9.6	10.3
Carbon dioxide (%)			
Range	8.8-9.9	8.1-9.8	8.4-9.8
Average	9.2	9.0	9.0
Carbon monoxide (ppm)			
Range	16-1980 ^c	16-1883 ^c	25-1860 ^c
Average	505	250	529
Total hydrocarbon (ppm) ^b			
Range	41-589 ^c	36-372 ^c	28-605 ^c
Average	74.6	45	61

^a Concentrations on dry gas basis.

^b Total HC as propane.

^c These readings represent the upper limits of detection. Actual values were probably higher.

TABLE I-21. CHLORIDE CONCENTRATION IN
SCRUBBER INLET AND OUTLET
WATER AND KILN ASH LIQUID
(g/L)

	Run 1	Run 2	Run 3
Inlet water	0.035	0.033	0.033
Outlet water ^a	32.5	39.5	49.5
Kiln ash liquid	28.3	26.0	40.8

^a pH of outlet water was 1 in all runs.

TABLE I-22. CONCENTRATIONS OF VOLATILE AND SEMIVOLATILE POHCs
AND PICs IN WATER SAMPLES

Compound	Concentration ($\mu\text{g/L}$) in given sample					
	In-flow water ^a			Out-flow water ^b		
	Run 1	Run 2	Run 3	Run 1	Run 2	Run 3
<u>Volatiles</u>						
Methylene chloride	< 1	40	4.8	< 1	2.2	< 1
Chloroform	100	100	85	< 1	< 1	< 1
1,1,1-Trichloroethane	< 1	< 1	< 1	< 1	< 1	< 1
Carbon tetrachloride	< 2	< 2	< 2	< 2	< 2	< 2
Trichloroethylene	< 1	< 1	< 1	< 1	< 1	< 1
Tetrachloroethylene	< 1	< 1	< 1	< 1	< 1	< 1
Toluene	1.7	2.1	3.7	13	< 1	< 1
cis-1,4-Dichloro-2-butene	< 1	< 1	< 1	< 1	< 1	< 1
Benzene	< 3	< 3	< 3	4.3	< 3	< 3
Chlorobenzene	< 1	< 1	< 1	< 1	< 1	< 1
Bromodichloromethane	29	27	21	< 1	< 1	< 1
Dibromochloromethane	12	11	8.7	< 1	< 1	< 1
Bromoform	< 2	< 2	< 2	< 2	< 2	< 2
<u>Semivolatiles</u>						
trans-1,4-Dichloro-2-butene	c	< 2	< 2	< 2	< 2	< 2
Phenol	-	2.0	9.6	3.9	< 1	6.6
Benzyl chloride	-	< 1	< 1	< 1	< 1	< 1
Hexachloroethane	-	< 3	< 3	< 3	< 3	< 3
Naphthalene	-	< 1	< 1	2.5	2.1	3.1

^a Average flow rate for the inflow water was 227 L/hr (62 gal/hr).

^b Average flow rate for the outflow water was 7,310 L/hr (2,010 gal/hr).

^c Sample lost.

METALS

All waste feed samples were analyzed for metals content and the results are presented in Table I-23. Barium, chromium, nickel, and lead were the most frequently detected metals, with nickel and lead appearing in the largest concentrations.

Both the sluice water used to carry the ash to the clarifier and the stack particulate were analyzed to determine metal content. The results of the sluice water analysis are presented in Table I-24. The metal emissions in the particulate are presented in Table I-25. Metals detected in the stack particulate samples include barium, chromium, nickel, lead, and selenium. The output rate in the particulate is compared with the input rate in Table I-26.

ANALYSIS OF WASTE FEEDS

A general analysis was performed on the waste feeds to determine chlorine, water, and ash content, as well as heating value. An ultimate analysis was performed to determine elemental content of the wastes. The viscosity of the liquid waste was also determined. The results are presented in Table I-27.

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (a) external auditing; (b) sampling QA activities; and (c) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings can be found in the final report (Appendix K).

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. Performance audits (quantitative evaluations) and systems audits (qualitative evaluations) were conducted for the analysis phases of the project.

Sampling Audit--

A stringent sampling audit consisting of performance and systems audits was conducted on site at the incinerator facility. Performance audits were conducted on:

- Meter boxes, Modified Method 5;
- Barometer, aneroid;
- Beckman NDIR;
- Orsat;
- RAC console temperature gauge; and
- Dry gas meter.

TABLE I-24. METAL CONCENTRATION IN
KILN ASH SLUICE WATER
SAMPLES (mg/L)

Metal	Run 1	Run 2	Run 3
Ag	< 19	< 19	22.1
As	< 130	< 130	130
Ba	4,240	671	779
Be	< 19	< 19	85
Cd	< 35	124	38
Cr	499	665	952
Hg	< 190	< 190	< 190
Ni	15,900	5,470	7,190
Pb	477	2,030	494
Sb	< 100	< 100	159
Se	< 200	< 200	689
Ti	< 300	< 300	807

TABLE I-25. METAL EMISSIONS IN
STACK EFFLUENT

Metal	Concentration in particulates ($\mu\text{g/g}$)		
	Run 1	Run 2	Run 3
Ag	648	1,880	222
As	< 5,000	< 17,000	< 7,000
Ba	648	3,090	742
Be	< 120	< 420	< 180
Cd	1,260	1,160	953
Cr	668	a	a
Hg	< 1,000	< 3,500	< 1,500
Ni	5,170	4,920	4,260
Pb	10,700	31,100	35,000
Sb	< 1,200	< 4,100	< 1,700
Se	13,800	61,600	28,300
Tl	< 900	< 3,100	< 1,300

Emission rate (g/min)			
Ag	0.0064	0.0054	0.0015
As	< 0.049	< 0.049	< 0.049
Ba	0.0064	0.089	0.0051
Be	< 0.0012	< 0.0012	< 0.0012
Cd	0.012	0.0033	0.0066
Cr	0.0066	a	a
Hg	< 0.010	< 0.010	< 0.010
Ni	0.051	0.014	0.030
Pb	0.11	0.089	0.24
Sb	< 0.012	< 0.012	< 0.012
Se	0.14	0.18	0.20
Tl	< 0.0089	< 0.0089	< 0.0089

^a High blank value.

TABLE I-26. METAL IN STACK EMISSIONS
AS PERCENT OF INPUT
IN WASTE FEEDS^a

Metal	Run 1	Run 2	Run 3
Ag	b	b	b
As	b	b	d
Ba	1.6	13.3	0.75
Be	b	b	b
Cd	3.1	0.94	2.4
Cr	0.43	c	c
Hg	b	b	b
Ni	0.28	0.091	0.25
Pb	4.2	1.2	1.8
Sb	d	d	d
Se	b,c	b,c	c
Tl	b	b	b

^a Percent = $\frac{\text{stack output, g/min}}{\text{waste input, g/min}} \times 100$

^b Amount in waste input was a < value.

^c High blank value for output.

^d Amount in stack output was a < value.

TABLE 1-27. ANALYSIS OF WASTE FEEDS

Waste	Run	Percent Cl	Percent H ₂ O	Percent ash	kJ/kg	Heating value (Btu/lb)	
Liquid waste	1	24.54	2.81	0.47	29,100	(12,500)	
	2 ^a	24.91	1.78	0.22	28,900	(12,400)	
	3	25.55	2.90	0.30	28,900	(12,400)	
Filter cake	1	8.26	55.12	17.25	8,700	(3,700)	
	2	9.79	51.41	17.14	8,400	(3,600)	
	3	9.19	56.40	17.95	8,000	(3,400)	
DCB coke	1	21.55	0.47	0.55	28,700	(12,400)	
	2	24.80	2.72	0.21	28,100	(12,100)	
	3	21.45	4.84	0.72	27,600	(11,900)	
ISOM coke	1	18.39	38.87	0.68	15,900	(6,900)	
	2	17.20	37.94	1.54	14,600	(6,300)	
	3	18.67	31.63	1.63	19,000	(8,200)	
Paint waste	1	0.050	0.59	1.37	32,400	(13,900)	
	2	0.18	0.43	2.31	31,800	(13,700)	
	3	0.14	0.51	8.16	30,700	(13,200)	
Ultimate analysis	Run	Percent Cl	Percent H	Percent N	Percent S	Percent ash	Percent O (by difference)
Liquid waste	2	27.25	7.13	0.42	0.009	0.36	2.69
Filter cake	2	12.18	7.81	0.43	0.002	18.00	43.73
DCB coke	2	26.70	4.55	0.022	0.009	0.32	1.48
ISOM coke	2	17.98	7.43	0.10	0.002	1.05	41.44
Paint waste	2	0.33	8.19	0.10	0.002	2.21	21.26

^a Liquid waste in Run 2 had a viscosity of 29.3 SUS at 100°F, and is believed to be representative of the liquid waste used in all three runs.

Systems audits were conducted on:

- Modified Method 5 train operation;
- Volatile organic sampling train operation;
- EPA Standard Methods 1, 2, 3, and 4;
- Process data collection;
- Scrubber effluent sampling;
- Waste feed grab sampling;
- Orsat operation; and
- Integrated gas bag sampling.

The results of the performance audits indicated that there were no problems which affected the quality of the sampling. The systems audit indicated no significant operational problems.

Laboratory Audit--

A performance audit and limited systems audit were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on GC/MS analysis of volatile and/or semivolatile organics.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of purgeable organics. The semivolatile results are variable but are acceptable.

Systems audits demonstrated that laboratory analyses were done according to protocol.

Sampling QA Activities

In general, sampling QA activities were scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures excluded spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure was to monitor the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

APPENDIX J

SUMMARY OF RESULTS FROM TESTING AT
THE TWI INCINERATOR

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SECTION 1

INTRODUCTION

The Environmental Protection Agency (EPA) is preparing a regulatory impact analysis (RIA) of hazardous waste incineration. Emissions data for the RIA are being provided by a sampling and analysis program. EPA selected for emission measurements the hazardous waste incinerator at Trade Waste Incineration, Inc., a commercial facility located in Illinois.

Test runs were conducted at this site during the period February 2 to 5, 1983. Samples of waste feeds and gaseous, liquid, and solid effluents were collected and analyzed. The following sections of this appendix present a summary of results, a brief process description, a synopsis of the test plan and sampling and analysis methods, and a discussion of the test results.

SECTION 2

SUMMARY OF RESULTS

A major objective of this test program was to identify and quantify principal organic hazardous constituents (POHCs) in the waste feeds, and the POHCs and products of incomplete combustion (PICs) in the stack gas from the incinerator facility. POHCs were also quantified in ash and scrubber effluents. Particulate emissions, HCl removal efficiency, and metal emissions were also determined.

The compounds identified as POHCs and PICs were divided into two groups: volatiles and semivolatiles. POHCs in the waste feeds and stack gases were:

Volatiles

Methylene chloride
Chloroform
Methylene bromide
1,1,1-Trichloroethane
Carbon tetrachloride
Trichloroethylene
Benzene
Tetrachloroethylene
Toluene
Chlorobenzene

Semivolatiles

Hexachlorocyclopentadiene
Bis-(2-ethylhexyl)-phthalate
Chlordane (Runs 1-3)
Naphthalene (Run 4)
Hexachlorobutadiene (Run 4)

PICs in the effluent gas were:

Volatiles

Bromochloromethane
Bromodichloromethane
Dibromochloromethane
Bromoform

Semivolatiles

Naphthalene (Runs 1-3)

The volatile POHCs which were found in the highest concentrations in both the feed and stack effluent were toluene and benzene. In this report, data on emissions of POHCs are presented as uncorrected concentrations and emission rates accompanied by results for all "blanks," expressed in the same units.

Results of the particulate emission measurements show that the average particulate concentration (corrected to 7% O₂) for the four runs was 168 mg/dscm (0.074 gr/dscf); below the EPA regulation for hazardous waste

incinerators of 180 mg/dscm (0.08 gr/dscf). Chloride emissions averaged 0.16 kg/hr (0.35 lb/hr) for the four runs, below the EPA maximum allowable emission rate of 1.8 kg/hr. Barium, chromium, and lead were the most frequently detected metals in the waste feed. The highest metal concentrations in the waste and stack particulate were for lead. The liquid organic waste contained about 0.1% lead and the stack particulate about 10% lead.

All of these results are presented in detail in Section 5. This appendix contains minimal interpretation of the results.

SECTION 3

PROCESS DESCRIPTION AND PROCESS DATA

The incinerator design features a two-stage combustion chamber, a quench section, a Venturi scrubber and a packed scrubber (see Figure J-1).

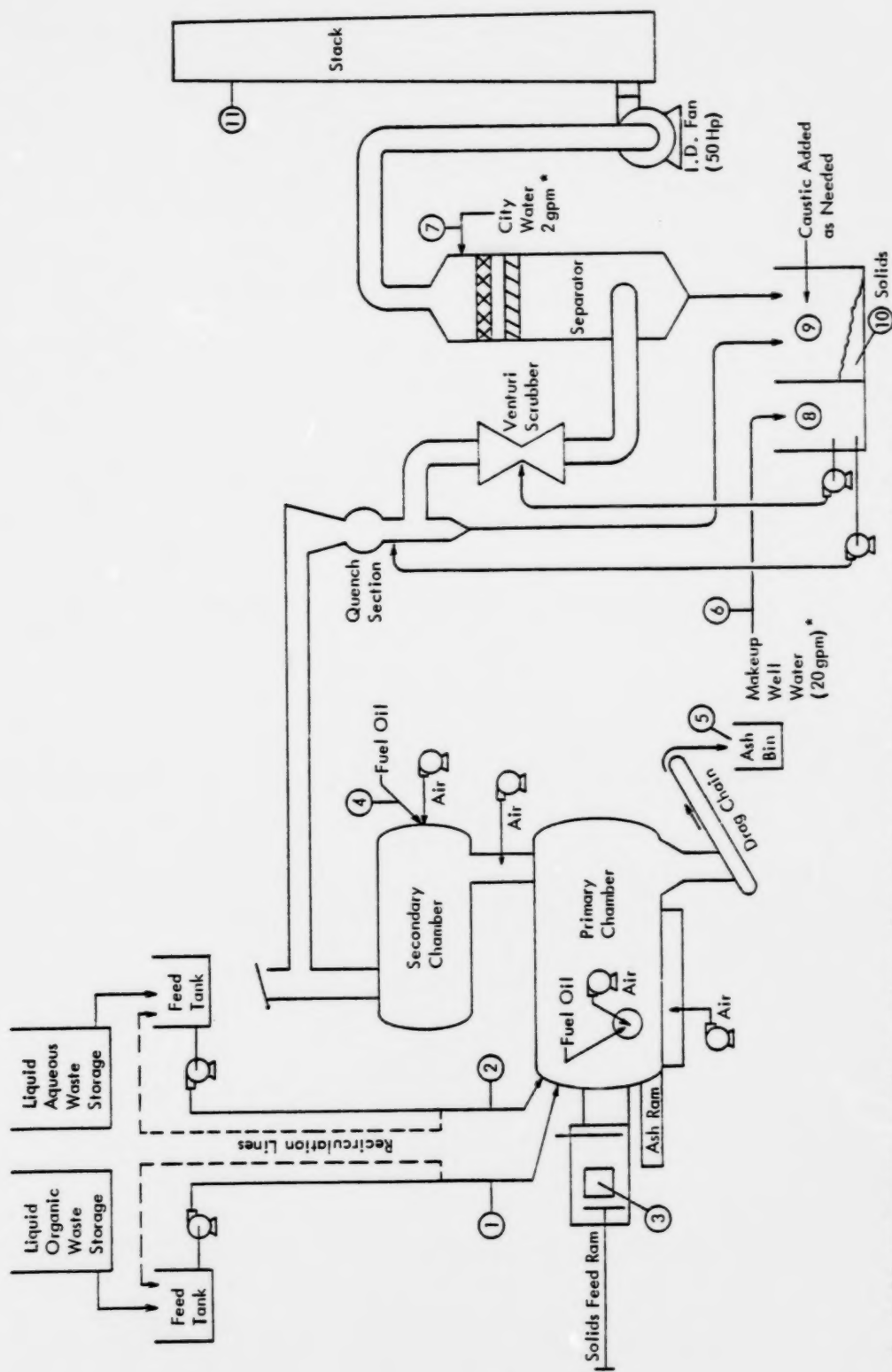
Liquid organic waste and aqueous waste are stored in separate 9,000-gal. storage tanks. These wastes are continuously fed into the primary combustion chamber through separate lines. Solid waste is inserted into the chamber using a ram at a rate of approximately one 45-lb batch every 5 min. Combustion air and fuel oil are also admitted into the chamber to control the combustion temperature. Ash is removed from the chamber periodically by using a ram to clear the ash from the floor of the chamber into an ash bin. A drawing of the combustion chambers, including thermocouple (T/C) locations, is presented in Figure J-2.

The combustion gases exit the primary combustion chamber and enter a secondary chamber where additional fuel oil and combustion air are admitted to maintain desired operating conditions. After leaving the second chamber, the gases are quenched and passed through a venturi scrubber for particulate and HCl removal. The gases then travel up through a mist eliminator and exit the process through the stack.

Water for the quench and the two scrubbers is recirculated. Makeup for these processes is provided by both well water and city water at a rate of approximately 70 L/min (20 gal/min) for the quench and 7 L/min (2 gal/min) for the demister following the scrubber. During Runs 1 and 2 city water was used for the quench makeup and well water was used for the demister makeup. During Runs 3 and 4 the well water was frozen and city water was used for all makeup purposes. Effluent waters are channeled into a settling tank where solids are allowed to settle out and caustic soda is added to neutralize the water before recirculating it through the quench and scrubber.

During the first three runs a solid waste made up of sand, gravel, and approximately 5% chlordane was fed to the chamber. During the fourth run a solid waste containing hexachlorocyclopentadiene (HCCP) and having a high Btu content was fed to the chamber. The same aqueous and organic wastes were burned during all four runs.

Process data for the four runs are summarized in Table J-1. These data show that the primary chamber temperature averaged 1130°C for the first three runs and dropped to 988°C for the fourth run. The secondary chamber temperature averaged 1100°C for the first three runs and dropped to 965°C for the fourth run.



* Well water was used as makeup for demisters in Runs 1 and 2. City water was used as makeup for the quench and scrubber in Runs 1 and 2 and for all makeup purposes in the remaining runs.

Figure J-1. Summary of sampling locations.

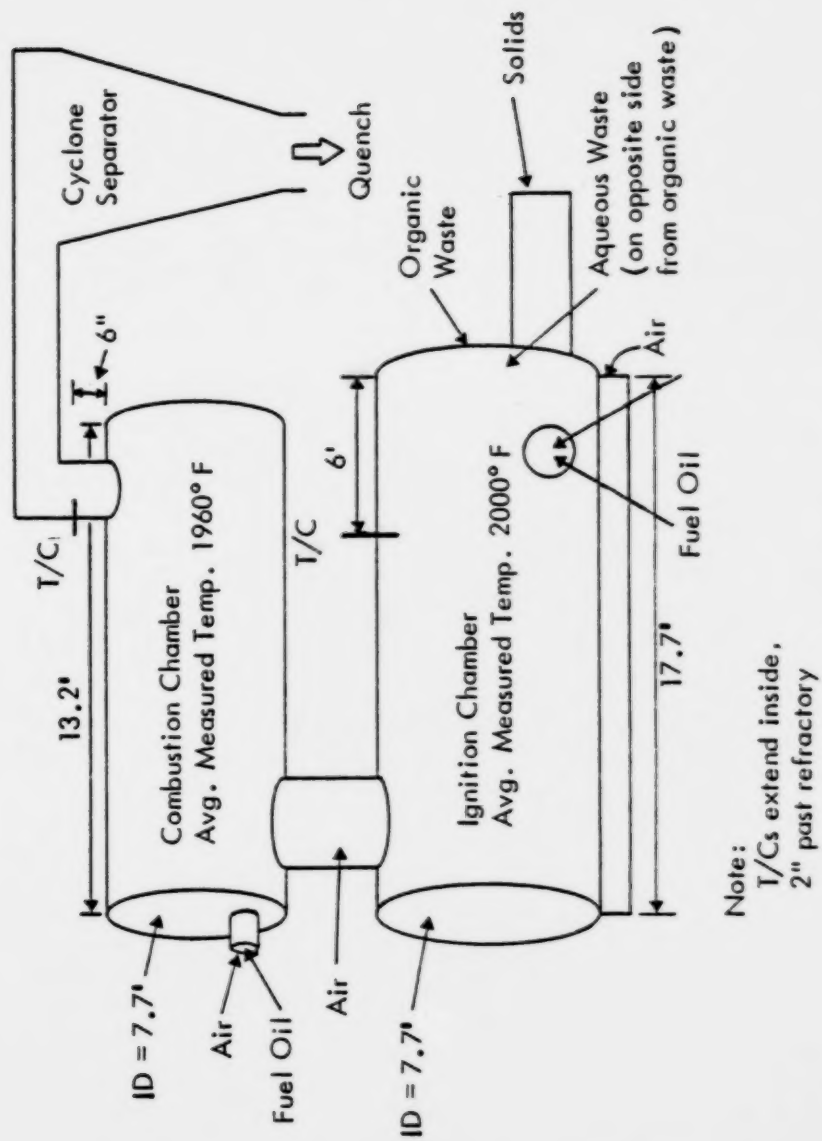


Figure J-2. Combustion chamber diagram.

TABLE J-1. PROCESS DATA (FROM PLANT INSTRUMENTS)

	Run 1	Run 2	Run 3	Run 4
Waste feed heat load, ^a 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	7.42 (7.30)	7.89 (7.48)	6.38 (6.05)	7.36 (6.98)
Auxiliary fuel heat load, 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	2.7 (2.6)	3.8 (3.6)	6.3 (6.0)	3.2 (3.0)
Primary chamber temp., °C ^b (°F)	1,140 (2,078)	1,110 (2,030)	1,130 (2,070)	988 (1,810)
Secondary chamber temp., °C ^b (°F)	1,110 (2,030)	1,090 (2,000)	1,110 (2,030)	965 (1,770)
Gas after prequench temp., °C (°F)	80 (176)	79 (174)	79 (174)	73 (163)
O ₂ , %	14.2	13.7	14.5	16.7
DP across venturi, kPa (in.wc)	2.0-2.2 (8-9)	1.7-2.0 (7-8)	1.7 (7)	2.0 (8)
DP across mist pads, kPa (in.wc)	1.8 (7.4)	1.8 (7.6)	1.9 (7.8)	1.7 (7.2)
H ₂ O flow to venturi, L/min (gpm)	210 (55)	c	190 (51)	200 (54)
Organic waste feed rate, L/min (gpm)	2.9 (0.78)	2.7 (0.71)	2.5 (0.67)	1.5 (0.39)
Aqueous waste feed rate, L/min (gpm)	d	1.5 (0.40)	2.6 (0.70)	2.2 (0.58)
Organic waste atomizing air pressure, kPa (psig)	69 (10)	100 (15)	100 (15)	210 (30)
Organic waste feed pressure, kPa (psig)	24 (3.5)	27 (3.9)	21 (3.1)	14 (2.0)
Aqueous waste feed pressure, kPa (psig)	24 (3.5)	21 (3.1)	21 (3.0)	23 (3.4)
Combustion chamber residence time, sec	4.7	3.5	3.5	3.0
Stack height, m (ft)	16 (52)	16 (52)	16 (52)	16 (52)
Stack velocity, m/min (ft/min)	876 (2,874)	1,014 (3,328)	1,035 (3,394)	1,071 (3,513)
Stack temperature, °C (°F)	66 (151)	59 (139)	62 (143)	52 (125)

^a Heat load calculations based on HHV of wastes; does not include auxiliary fuel.

^b Temperature as measured by thermocouples that projected into gas stream. Thermocouples even with refractory were not used because it was determined that they had been replaced with wrong type of thermocouple wire.

^c Line frozen during Run 2.

^d Flow meter not yet installed.

The combined residence time in the combustion chambers ranged from 3.0 to 4.7 sec. The average heat load to the incinerator, based on waste feeds and auxiliary fuel, was 11.0×10^6 kJ/hr.

Waste feed and auxiliary fuel oil flow rates for the four runs are listed in Table J-2.

TABLE J-2. WASTE FEED RATES,^a kg/min (lb/min)

	Run 1	Run 2	Run 3	Run 4
Aqueous waste	8.10 (17.8)	5.06 (11.2)	3.83 (8.44)	2.55 (5.61)
Liquid organic waste ^b	3.17 (6.91)	3.24 (7.14)	2.60 (5.74)	1.05 (2.32)
Solid waste				
Sand/gravel	3.89	4.43	4.03	-
with chlordane	(8.58)	(9.76)	(8.88)	
High Btu solids with C-56	-	-	-	4.05 (8.92)
Fuel oil	1.0 (2.2)	1.4 (3.1)	2.5 (5.2)	1.2 (2.6)

^a Feed rates based on change in waste feed storage tank levels.

^b Organic feed rates calculated from volumetric flow rates using an average density of 0.923 kg/L for the waste.

SECTION 4

TEST PLAN AND SAMPLING AND ANALYSIS METHODS

A description of the sampling method and the subsequent analysis performed on the samples is provided in Table J-3 for each sampling point shown in Figure J-1. All collected samples that were not analyzed and the portions of samples remaining after analysis were archived.

Aqueous and organic waste streams were fed continuously to the combustion chamber and sampled at 15-min intervals for the duration of the run. These samples were used to generate a composite sample for each waste. Solid waste was admitted in 45-lb batches during Runs 1 through 4. These batches were weighed and a grab sample was taken from each batch and then used to generate a composite for each run. A 50/50 mixture (by volume) of carbon tetrachloride and trichloroethylene was continuously pumped (spiked) into the organic waste feed line, at a rate of about 40 ml/min. An in-line mixer was installed by the plant between the connection of the spike pump to the feed line and the sample tap.

The method of sampling effluents consisted of grab sampling for the kiln ash, makeup and effluent waters, and the solids in the water settling tank. Stack sampling was accomplished using a Modified Method 5 (MM5) train for particulates, chlorides, metals, and semivolatile POHCs. Two methods were used for sampling volatile POHCs; gas bags and a Volatile Organic Sampling Train (VOST). Continuous monitors were used for O₂, CO₂, CO and total hydrocarbons. A schematic diagram of the MM5 train is shown in Figure J-3, and a diagram of the VOST system is shown in Figure J-4.

Gas bag samples were collected in 15-L Tedlar bags, drawing gas into the bags over a period of about 1 hr. Gas bags filled with prepure N₂ in the field were also prepared as gas bag blanks.

In addition to gas bags, the two VOST sampling systems were also used to sample volatile POHCs. The VOST system consists of a Tenax trap and a backup Tenax/charcoal trap. For fast VOST gas was drawn through the two traps, in series, at a rate of 1 L/min for 20 min. The first pair of traps was then removed and replaced with another pair, until a total of six pairs were collected (i.e., 120 min of sampling time). Slow VOST was used in addition to fast VOST for Runs 1 through 3. Slow VOST was similar to fast VOST but the sampling rate was slower (0.25 L/min) and the sampling time was longer (40 min). Only two pairs of traps were used. The first pair was in the stack during the same times as the first and third fast VOST pairs. The second slow VOST pair was in the stack during the same times as the fifth and sixth fast VOST pairs.

TABLE J-3. SUMMARY OF SAMPLING AND ANALYSIS

Sampling point	Number of samples per run	Sampling method or procedure	Analysis
<u>Feed Samples</u>			
① Liquid organic (spiked)	1 Composite	Grab sample every 15 min	HHV, ash, viscosity Cl, H ₂ O, ultimate Metals POHCs
	3 subsamples	Grab samples	
② Aqueous	1 Composite	Grab sample every 15 min	HHV, ash Cl, H ₂ O, Br POHCs
③ Solids	1 Composite	Grab sample of every batch (~ 10 per hour)	HHV, ash Cl, H ₂ O Metals POHCs
④ Fuel oil	1 Grab	1 Grab sample	Volatile POHCs Archive
<u>Ash Samples</u>			
⑤ Ash	1 Composite	1 Grab whenever ash discharge is operated (5 to 6 times per hour)	Metals EP toxicity POHCs
<u>Water Samples</u>			
⑥ Well water	1 Grab	1 Grab sample	Volatile POHCs Archive
⑦ City water	1 Grab	1 Grab sample	Volatile POHCs Archive
⑧ Recirc. H ₂ O	1 Composite	3 Grab samples	Volatile POHCs pH Archive

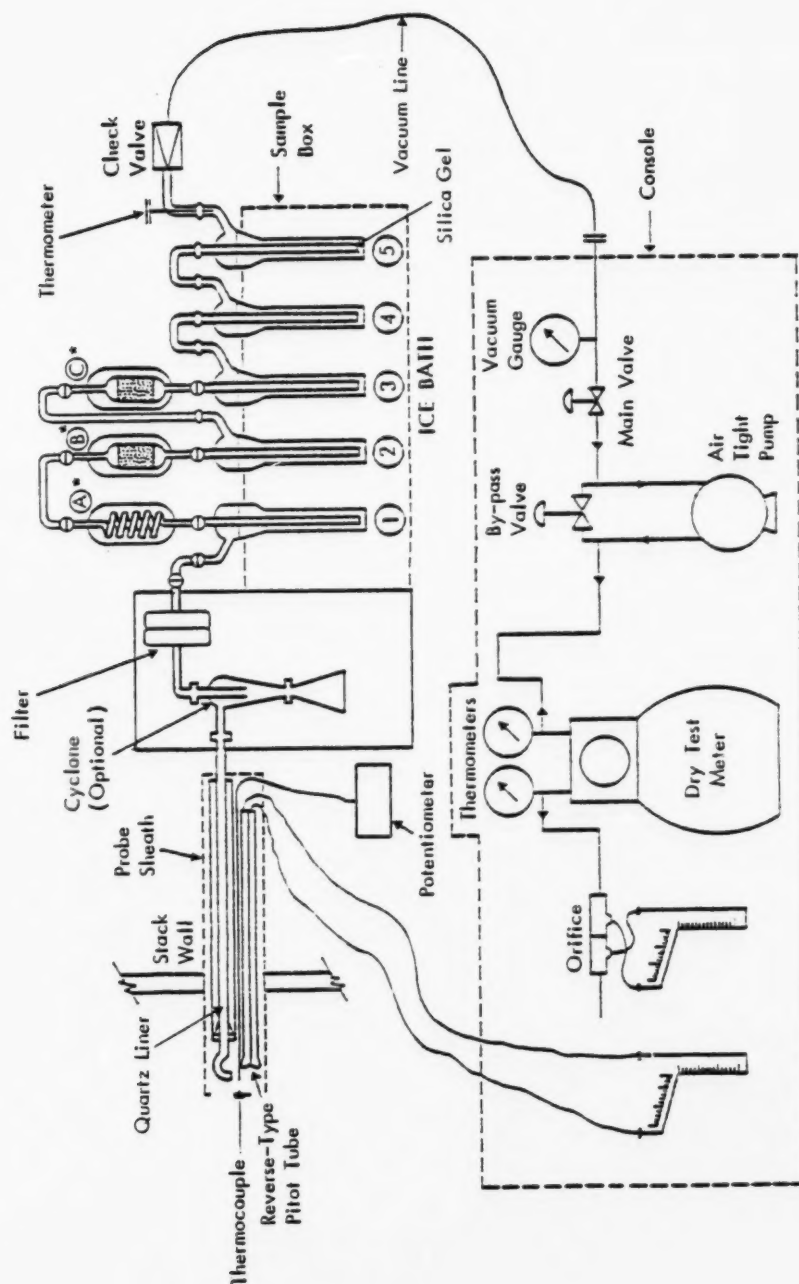
(continued)

TABLE J-3. (continued)

Sampling point	Number of samples per run	Sampling method or procedure	Analysis
<u>Water Samples (concluded)</u>			
⑨ Return H ₂ O	1 Composite	3 Grab samples	Volatile POHCs pH Archive
⑩ Solids in H ₂ O Recirc. tank	^a	Grab sample	Metals POHCs
<u>Stack</u>			
⑪ MM5 train	1	MM5 train	Particulate weight and metals in particulate Semivolatile POHCs and PICs (front half and back half) Cl
	2 Pair of traps	Slow VOST	Volatile POHCs and PICs
	6 Pair of traps	Fast VOST	Volatile POHCs and PICs ^b
	1	Integrated gas bag	Volatile POHCs and PICs
	1	Integrated gas bag	CO ₂ and O ₂ by Orsat
	Cont.	Continuous monitors	CO, CO ₂ , O ₂ , HC

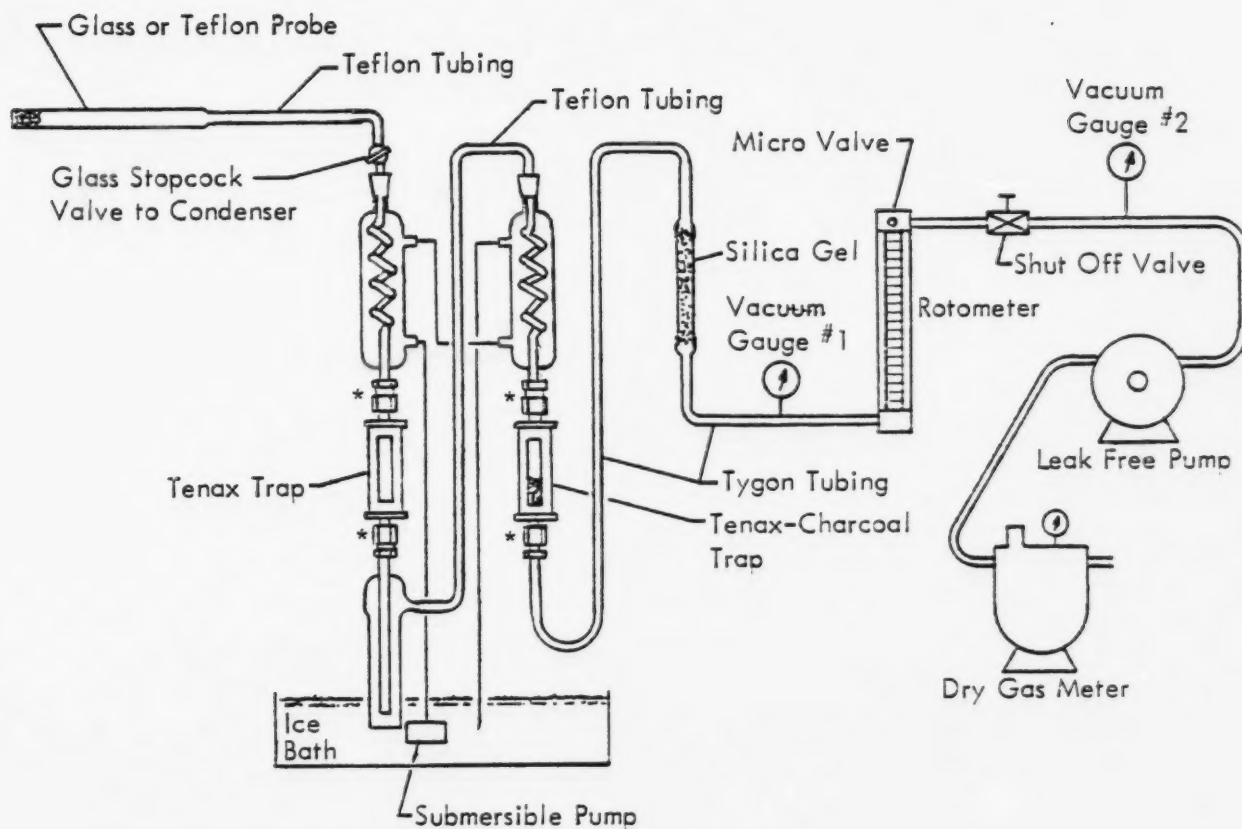
^a One grab sample is to be taken after Run 3.

^b Four pair of traps were analyzed.



- | | |
|--|--------------------------|
| ① Modified Greenburg-Smith, Reversed, Empty | Ⓐ Condenser |
| ② Greenburg-Smith, 50 ml of Double Distilled in Glass H ₂ O | Ⓑ XAD Resin Cartridge #1 |
| ③ Greenburg-Smith, 100 ml of 0.1 N KOH | Ⓒ XAD Resin Cartridge #2 |
| ④ Modified Greenburg-Smith Empty | * Ice Water Jacket |
| ⑤ Modified Greenburg-Smith, SiO ₂ | |

Figure J-3. Modified Method 5 sampling train (NM5).



* Special O-Ringed Stainless Steel Fittings

Figure J-4. Volatile organic sampling train (VOST).

Field blank sample pairs were exposed to the sampling location for the same period required to insert sample traps into the VOST apparatus. All traps were then returned to the analytical laboratory for analysis of volatile POHCs.

POHCs in the waste feed and stack samples were determined by gas chromatography/mass spectrometry (GC/MS). Prior to analysis, the waste feed samples were homogenized either manually or mechanically. The homogenized feed samples were analyzed for volatile POHCs using a protocol based on Method A101b in the A. D. Little report, "Sampling and Analysis Methods for Hazardous Waste Incineration." A 2.0-g aliquot of the mixed waste feed sample was dispersed in 40 mL of prepurified tetraglyme. A 10- μ L aliquot of the tetraglyme solution was spiked into 5.0 mL of reagent water along with 100 ng each of a three-component internal standard (equivalent to 200 μ g/g each in the original sample). The sample was then analyzed by the purge-trap-desorb technique described in Method A101a in the A. D. Little report (EPA Method 624). Depending on the levels of volatile POHCs in the sample, the analysis was repeated using a larger aliquot (up to 250 μ L) or a diluted aliquot of the tetraglyme extract.

The semivolatile POHCs were analyzed in the waste feed samples according to Method P023 in the A. D. Little report. A 5-g portion of the homogenized waste feed sample was spiked with semivolatile surrogate compounds such as decafluorobiphenyl, D₈-naphthalene, and D₁₂-chrysene at a level of 250 to 1,000 μ g/g, depending on the expected sample complexity. The sample was allowed to equilibrate with the spiked compounds and then was extracted with 50 mL of methylene chloride using an ultrasonic probe. If an aqueous phase appeared on the surface of the methylene chloride, the pH was checked and the sample neutralized, if necessary. The extract was then shaken, dried with anhydrous sodium sulfate, sonified for an additional 2 min, diluted to 150 mL with methylene chloride, and mixed.

An aliquot of the extract was then screened by gas chromatography (GC) with a flame ionization detector. If necessary, the extract was further diluted or concentrated to obtain a sample with an appropriate concentration for analysis by GC/MS on a fused silica capillary column. A 1.0-mL aliquot of the extract was then spiked with 20 μ g of D₁₀-anthracene internal standard and analyzed by fused silica capillary column GC/MS.

The volatile POHCs from the stack gas samples were contained in Tedlar gas bags and on the Tenax and Tenax/charcoal traps used with VOST. A known amount of the gas bag sample, usually 5.0 L, was transferred onto a Tenax/charcoal trap (the same type of trap used as the backup trap in the VOST). To make the transfer a needle was inserted into the bag and the contents drawn onto the trap at a rate of 500 mL/min for 10 min. These traps were then analyzed using the same techniques that are described below for the VOST traps.

Prior to GC/MS analysis, all Tenax and Tenax/charcoal adsorbent trap samples, transferred bag samples, and calibration standards were spiked with 50 ng of perfluorobenzene, D₄-1,2-dichloroethane, and bromofluorobenzene internal standards using the flash vaporization technique, in which the spiking solution was vaporized and carried onto the trap with a carrier gas.

In order to accomplish this spiking, the glass traps were attached to the injection port (160°C) of a GC column. Helium flow, set to about 50 mL/min, was turned on through the trap and a syringe needle injected through the GC septum port. Slowly, 1 µL of a 25-ng/µL methanolic solution of the internal standards was expelled from the syringe over about a 15-sec period. At the end of about 25 sec, the gas flow through the trap was shut off and the syringe removed. The total flow of gas through the traps during spiking was only about 25 mL.

To analyze the traps, the contents of the wet traps (dry traps in the case of method blanks, field blanks, calibration standards, and gas bag samples) were thermally desorbed using a stream of carrier gas into a water column (1 to 5 mL), which is a component of the EPA Method 624 purge-trap-desorb GC/MS analysis system. The traps were desorbed at a flow rate of 100 mL/min for 10 min at 180°C. The desorbed compounds passed into the bottom of the water column, were purged from the water, and then were collected on an analytical adsorbent trap also containing Tenax and charcoal. The compounds were then desorbed from the analytical adsorbent trap into the GC/MS system per EPA Method 624.

Analysis of semivolatile POHCs in the stack involved several components of the MM5 train. Each of the components of the MM5 train was spiked with a separate surrogate standard according to the following scheme:

Front XAD-2 resin trap - 20 µg of ¹³C-tetrachlorobiphenyl
Back XAD-2 resin trap - 20 µg of decafluorobiphenyl
Particulate filter plus probe rinse residue - 20 µg of D₁₂-chrysene
Condensate water - 20 µg of D₈-naphthalene

The spiked components were normally extracted separately and the extracts combined, dried with anhydrous sodium sulfate, concentrated to 1.0 mL using Kuderna-Danish evaporation according to Method P031 in the A. D. Little report and using supplementary gentle nitrogen blowdown, and analyzed by fused silica capillary column GC/MS. The samples were extracted according to the following procedures:

XAD-2 resin traps: The XAD-2 resin traps were Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Particulate filters plus probe rinse residues: The residue from the acetone rinse of the probe was added to the particulate and Soxhlet-extracted with methylene chloride for 16 hr as described in Method P024b in the A. D. Little report.

Condensate water: The condensate water was extracted with three 60-mL aliquots of methylene chloride as described in Method P021a in the A. D. Little report (equivalent to EPA Method 625).

POHCs in scrubber water and ash samples were analyzed using the procedures described above. To analyze for metals, the samples were acid digested and analyzed by inductively coupled argon plasma emission spectrophotometry and/or atomic absorption spectrophotometry. Particulate and

chloride analyses were conducted according to EPA Method 5 and EPA Method 325.2, respectively, from "Methods for Chemical Analysis of Water and Wastes." EP toxicity tests were conducted according to Federal Register, 45, May 19, 1980. Water content in the waste feeds was determined by the Karl Fischer titration method. Chlorine in the waste feed and chamber ash was determined by potentiometric titration using a Fisher Titralizer Model 41. To determine bromine content, waste feed samples were combusted in a Shoniger flask, the bromine oxidized to bromate with hypochlorite, and the excess hypochlorite destroyed upon acidification with sulfuric acid. Potassium iodide was then added and the liberated iodine was titrated with thio-sulfate. Heating value was determined by ASTM Methods D2015-66 and D240-64. Orsat analyses were performed according to EPA Method 3. Continuous monitoring involved the following:

- O₂ - Beckman Model 742 (polarographic sensor)
- CO - Beckman Model 215A (nondispersive infrared)
- HC - Beckman Model 402 (heated oven flame ionization detector)
- CO₂ - Horiba Model PIR-2000S (nondispersive infrared)

In Appendix B, descriptions of all the above sampling and analysis methods are presented in more detail.

SECTION 5

DISCUSSION OF RESULTS

INPUT AND OUTPUT CONCENTRATIONS AND RATES FOR POHCs

POHC input and output concentrations and mass rates were determined for all POHCs listed by EPA which were detected in the waste feeds at concentrations above 100 $\mu\text{g/g}$. No attempt was made to restrict the number of POHCs based on any selection or ranking method. All POHCs reported are compounds that were quantified against authentic standards. In all tables, values marked < indicate detection levels.

The concentration of volatile and semivolatile POHCs in the waste feeds and fuel oil are given in Tables J-4 and J-5, respectively. Table J-6 presents input rates for the POHCs, which were calculated based on the concentration values in Tables J-4 and J-5 and the waste feed rates in Table J-3.

Tables J-7 and J-8 present volatile POHC output data (not blank corrected) for gas bags, slow VOST and fast VOST samples. Table J-7 lists the output concentration in nanograms per liter and Table J-8 shows the output rates in grams per minute. The concentration of chloroform in the stack gases could be contributed to by the stripping of chloroform from the quench and scrubber makeup waters. This will be discussed later in this section.

Table J-9 presents values for semivolatile POHC emission concentrations and output rates (not blank corrected). Naphthalene was treated as a POHC in Run 4, due to its presence in the waste feed in concentrations greater than 100 $\mu\text{g/g}$. In Runs 1 through 3 it was treated as a PIC.

EMISSION OF PICs

Analysis of samples included identification of PICs, i.e., those compounds identified in the stack samples that are listed as POHCs but which were not found in the waste feed samples at concentrations above 100 $\mu\text{g/g}$. The output concentration and rate for volatile PICs are tabulated in Tables J-10 and J-11. Semivolatile PIC concentrations and emission rates are found in Table J-12. The concentration of bromodichloromethane and dibromochloromethane could be contributed to by the stripping of these compounds from the scrubber and quench waters. This will be discussed in a later section.

TABLE J-6. CONCENTRATION OF VOLATILE FORCS IN WASTE FEEDS (ug/g)

Compound	Run No.	Liquid organic waste				Liquid aqueous waste				Solid waste				Fuel oil
		1	2	3	4	1	2	3	4	1	2	3	4	
Methylene chloride		160	110	340	220	< 10	44	60	51	< 10	< 10	< 10	< 10	440
Chloroform		100	110	60	450	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	23
Methylene bromide		< 10	< 10	< 10	< 10	460	3,100	4,700	4,800	< 10	< 10	< 10	< 10	210
1,1,1-Trichloroethane		380	330	230	120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	220
Carbon tetrachloride		9,400	9,100	11,300	28,000	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Trichloroethylene		8,500	8,300	10,300	25,400	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	20
Benzene		72,000	46,000	58,000	65,000	< 10	< 10	< 10	< 10	< 10	74	< 10	4,300	91
Tetrachloroethylene		150	87	150	120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	200	380
Toluene		390,000	160,000	390,000	330,000	< 10	22	20	22	140	< 10	< 10	29,000	1,500
Chlorobenzene		410	390	500	340	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10

TABLE J-5. CONCENTRATION OF SEMIVOLATILE POHCs IN WASTE FEEDS^a (µg/g)

	Liquid organic waste				Solid waste			
	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4
Hexachlorocyclopentadiene	< 10	< 10	< 10	< 10	260	230	260	16,000
Bis-(2-ethyl hexyl)-phthalate	210	200	230	190	< 10	< 10	< 10	< 30
Chlordane	< 60	< 60	< 60	< 60	18,000	19,000	19,000	< 160
Naphthalene	33	38	33	23	< 10	< 10	< 10	7,100
Hexachlorobutadiene	< 10	< 10	< 10	< 10	< 10	< 10	< 10	280

^a Liquid aqueous waste and fuel oil were only analyzed for volatile POHCs. They were not expected to contain semivolatile POHCs.

TABLE J-6. TWI POHC INPUT RATES^a (g/min)

	Run 1	Run 2	Run 3	Run 4 ^b
<u>Volatiles</u>				
Methylene chloride	0.95	0.97	2.2	0.89
Chloroform	0.34	0.36	0.21	0.50
Methylene bromide	3.7	16	18	12
1,1,1-Trichloroethane	1.2	1.4	1.1	0.39
Carbon tetrachloride	30	29	29	29
Trichloroethylene	27	27	27	27
Benzene	230	150	150	68
Tetrachloroethylene	0.86	0.81	1.3	1.4
Toluene	1,200	520	1,000	460
Chlorobenzene	1.3	1.3	1.0	0.36
<u>Semivolatiles</u>				
Hexachlorocyclopentadiene	1.0	1.0	1.0	53
Bis-(2-ethyl hexyl)-phthalate	0.65	0.65	0.60	0.20
Chlordane	70	84	77	< 0.1
Naphthalene	< 0.1	< 0.1	< 0.1	29
Hexachlorobutadiene	< 0.1	< 0.1	< 0.1	1.1

^a These input rates include POHCs which were found in the fuel oil.

^b Run 4 used a different solid waste feed than Runs 1 through 3.

TABLE J-7. CONCENTRATION OF VOLATILE POHCs IN STACK EFFLUENT
(NOT BLANK CORRECTED), ng/L

	Gas ^a bag	Run 1						Slow VOST		Avg.
		Fast VOST					1st pair	2nd ^c pair		
		1st pair	3rd pair	5th pair	6th pair	Avg.				
Methylene chloride	64	24	4.5	3.6	1.7	8.5	64	74	-	
Chloroform	68	2.3	2.8	2.3	0.9	2.1	63	2.1	-	
Methylene bromide	1.5	1.4	0.2	0.2	< 0.1	0.4	2.1	3.9	-	
1,1,1-Trichloroethane	28	8.0	3.2	2.4	1.8	3.8	22	25	-	
Carbon tetrachloride	1.8	16	1.0	0.8	0.5	4.6	11	11	-	
Trichloroethylene	20 ^f	30 ^f	3.0	3.7	1.8	9.2	61 ^{b,f}	18 ^{b,f}	-	
Benzene	80	110 ^f	14	11	5.2	35	> 75 ^{b,f}	> 120 ^{b,f}	-	
Tetrachloroethylene	22 ^f	6 ^f	1.8	2.1	1.2	2.8	17 ^f	33 ^{b,f}	-	
Toluene	260 ^f	180 ^f	29	28	4.0	60	160 ^f	> 210 ^{b,f}	-	
Chlorobenzene	66	11	2.8	3.0	0.7	4.4	42	39	-	

(continued)

TABLE J-7. (continued)

Run 2											
	Gas bag	Gas bag ^a	Fast VOST				Slow VOST				
			1st pair	3rd pair	5th pair	6th pair	Avg.	1st pair	2nd pair	Avg.	
5 Methylene chloride	18	34	4.2	20	26	35	21	4.6	7.5	6.1	
26 Chloroform	3.9	20	68	35	41	42	46	50	84	67	
Methylene bromide	< 0.5	1.0	4.8	5.4	3.7	5.9	4.9	5.6	6.1	5.8	
1,1,1-Trichloroethane	4.8	16	20	21	16	18	19	26	29	28	
Carbon tetrachloride	< 1.0	< 1.0	12	1.5	0.7	0.6	3.7	4.7	2.4	3.6	
Trichloroethylene	29	18	19 ^f	9.5	6.8	7.3	11	11 ^f	13	12	
Benzene	8	70	110 ^f	130 ^f	110 ^f	100	110	130 ^f	6.8	70	
Tetrachloroethylene	3.4	10	19 ^f	17 ^f	14 ^f	11 ^f	15	17 ^f	15	16	
Toluene	18	160	450 ^f	240 ^f	520 ^f	160 ^f	340	130 ^f	130 ^f	130	
Chlorobenzene	< 0.5	13	42	22	35	10	27	30	16	23	

(continued)

TABLE J-7. (continued)

Run 3									
	Gas bag ^a	Fast VOST				Slow VOST			
		1st	3rd	5th	6th	1st	2nd	Avg.	
		pair	pair	pair	pair	pair	pair		
Methylene chloride	18	19	21	19	19	19	20	24	22
Chloroform	4.7	23	34	38	35	32	39	38	38
Methylene bromide	6.3	36	61	50	35	46	30	32	31
1,1,1-Trichloroethane	4.3	11	14	9.8	7.5	11	15	11	13
Carbon tetrachloride	1.4	5.1	2.2	2.1	1.3	2.7	3.4	2.1	2.8
Trichloroethylene	13	11 _f	4.8	5.0	4.2	6.2	5.3	4.3	5.8
Benzene	40	170 _f	180 _f	180 _f	160 _f	170	180 _f	11	100
Tetrachloroethylene	6.3	8 _f	13 _f	10 _f	10 _f	10	11 _b	12 _b	12
Toluene	77	400	500	64 _f	70 _f	340	> 85 _b	> 85 _b	-
Chlorobenzene	1.0	5.4	3.2	1.6	2.2	3.1	4.7	3.6	4.1

(continued)

TABLE J-7. (continued)

	Gas bag	Run 4 ^{d,e}					Avg.
		Fast VOST					
		2nd pair	4th pair	5th pair	6th pair		
Methylene chloride	50	71	5.8	5.2	3.8	21	
Chloroform	15	6.3	8.3	6.5	6.1	6.8	
Methylene bromide	23	11	14	12	13	12	
1,1,1-Trichloroethane	11	4.8	7.0	3.9	5.1	5.1	
Carbon Tetrachloride	2.1	0.4	4.0	2.0	0.4	1.7	
Trichloroethylene	10	2.0	1 _f 8	1.8	1.3	1.7	
Benzene	77	37	66 _f	42	50	49	
Tetrachloroethylene	5 _f 3	1 _f 6	0.1	1.9	2.4	1.4	
Toluene	430	150	310 _f	190 _f	220 _f	220	
Chlorobenzene	2.2	0.3	1.4	0.7	0.6	0.8	

^a This sample was a grab sample.

^b Sample saturated mass spectrometer.

^c The back trap of this pair of traps was not evaluated. These numbers are based on the front trap only.

^d The first and third pairs were not evaluated, the second and fourth pairs were used in their place.

^e Only fast VOST was used in Run 4.

^f The quantification of these numbers was based on a minor ion in the GC/MS, and therefore they are not as precise as the other numbers presented.

TABLE J-8. EMISSION RATE OF VOLATILE FORCS IN STACK EFFLUENT
(NOT BLANK CORRECTED), g/min

Gas bag ^a	Run 1						Slow VOST		
	Fast			VOST			1st pair	2nd ^b pair	Avg.
	1st pair	3rd pair	5th pair	6th pair	Avg.				
Methylene chloride	0.0026	0.00049	0.00038	0.00019	0.00092		0.0069	0.0080	-
Chloroform	0.0025	0.00030	0.00025	0.000096	0.00022		0.0068	0.0023	-
Methylene bromide	0.0015	0.00023	0.00019	< 0.00001	0.00005		0.0022	0.0042	-
1,1,1-Trichloroethane	0.0087	0.00026	0.00019	0.00019	0.00042		0.0024	0.0027	-
Carbon tetrachloride	0.0017	0.00011	0.000092	0.000052	0.00049		0.0011	0.0011	-
Trichloroethylene	0.0033	0.00032	0.00040	0.00020	0.0010		0.0066 ^{b, f}	0.0019 ^{b, f}	-
Benzene	0.012	0.0016	0.0012	0.00056	0.004		> 0.0081 ^{b, f}	> 0.013 ^{b, f}	-
Tetrachloroethylene	0.0067	0.00019	0.00022	0.00012	0.00030		0.0018	0.0036 ^f	-
Toluene	0.019	0.0032	0.0031	0.00044	0.006		0.017	> 0.023 ^f	-
Chlorobenzene	0.0012	0.00030	0.00032	0.000079	0.0006		0.0046	0.0043	-

(continued)

TABLE J-8. (continued)

Run 2										
Gas bag	Gas bag ^a	Fast VOST			Slow VOST			Avg.		
		1st pair	3rd pair	5th pair	1st pair	2nd pair	Avg.			
Methylene chloride	0.0025	0.0048	0.00060	0.0029	0.0037	0.0050	0.0044	0.00066	0.0011	0.0012
Chloroform	0.00056	0.0029	0.0097	0.0050	0.0060	0.0060	0.0065	0.0072	0.012	0.009
Methylene bromide	< 0.00007	0.00015	0.00069	0.00077	0.00052	0.00084	0.00070	0.00080	0.00087	0.00084
1,1,1-Trichloroethane	0.00069	0.0023	0.0029	0.0031	0.0022	0.0026	0.0027	0.0037	0.0041	0.0039
Carbon tetrachloride	< 0.0001	< 0.0001	0.0016	0.00022	0.000099	0.000092	0.0005	0.00067	0.00034	0.00050
Trichloroethylene	0.0041	0.0026	0.0028	0.0014	0.00097	0.0010	0.0016	0.0016	0.0019	0.0018
Benzene	0.0011	0.010	0.016	0.018	0.016	0.014	0.016	0.018	0.00097	0.010
Tetrachloroethylene	0.00049	0.0014	0.0027	0.0025	0.0020	0.0016	0.0022	0.0023	0.0022	0.0022
Toluene	0.0025	0.022	0.064	0.034	0.074	0.023	0.049	0.019	0.019	0.019
Chlorobenzene	< 0.00007	0.0018	0.0060	0.0031	0.0050	0.0015	0.0039	0.0042	0.0023	0.0032

(continued)

(continued)

TABLE J-8. (continued)

Gas bag	Run 3						
	Fast VOST			Slow VOST			Avg.
	1st pair	3rd pair	5th pair	6th pair	1st pair	2nd pair	
Methylene chloride	0.0026	0.0027	0.0030	0.0027	0.0027	0.0029	0.0032
Chloroform	0.00067	0.0033	0.0049	0.0055	0.0051	0.0056	0.0055
Methylene bromide	0.00091	0.0051	0.0088	0.0072	0.0051	0.0043	0.0045
1,1,1-Trichloroethane	0.00062	0.0016	0.0020	0.0014	0.0011	0.0022	0.0019
Carbon tetrachloride	0.00020	0.00074	0.00032	0.00030	0.00019	0.00049	0.00040
Trichloroethylene	0.0018	0.0016	0.00069	0.00072	0.00060	0.00077	0.00070
Benzene	0.0058	0.025	0.026	0.026	0.023	0.026	0.014
Tetrachloroethylene	0.00091	0.0012	0.0019	0.0015	0.0015	0.0016	0.0016
Toluene	0.011	0.058	0.072	0.0092	0.010	> 0.012	-
Chlorobenzene	0.00014	0.00077	0.00046	0.00023	0.00032	0.00068	0.00060

(continued)

TABLE J-8. (continued)

	Gas bag	Run 4 ^c				Avg.
		2nd pair	4th pair	Fast VOST ^e 5th pair	6th pair	
Methylene chloride	0.0084	0.012	0.00098	0.00088	0.00063	0.004
Chloroform	0.0025	0.0011	0.0014	0.0011	0.0010	0.0012
Methylene bromide	0.0038	0.0019	0.0023	0.0020	0.0022	0.0021
1,1,1-Trichloroethane	0.0019	0.00081	0.0012	0.00067	0.00086	0.00089
Carbon tetrachloride	0.0035	0.00068	0.00068	0.00033	0.00067	0.00044
Trichloroethylene	0.0017	0.00034	0.00030	0.00030	0.00022	0.00030
Benzene	0.013	0.0062	0.011	0.0071	0.0084	0.0083
Tetrachloroethylene	0.00090	0.00027	0.00020	0.00032	0.00040	0.00025
Toluene	0.073	0.025	0.052	0.032	0.037	0.037
Chlorobenzene	0.00037	0.000046	0.00024	0.00012	0.00010	0.00013

^a This sample was a grab sample.

^b This sample based on the first half of the pair of traps only.

^c No slow VOST in Run 4.

^d This sample saturated the mass spectrometer.

^e The first and third pairs were not analyzed. The second and fourth pairs were analyzed in their place.

^f The quantification of these numbers was based on a minor ion in the GC/MS, and therefore they are not as precise as the other numbers presented.

TABLE J-9. EMISSIONS OF SEMIVOLATILE POHCs (NOT BLANK CORRECTED)

Compound		Emission concentration (ng/L)			
		Run 1	Run 2	Run 3	Run 4
Hexachlorocyclopentadiene	front half	< 1	< 1	< 1	< 1
	back half	< 1	< 1	< 1	< 0.5
Bis-(2-ethyl hexyl)-phthalate	front half	2.8	1.4	2.6	1.5
	back half	3.4	2.8	2.2	2.0
Chlordane	front half	< 1	< 1	< 1	a
	back half	< 1	< 1	< 1	
Naphthalene	front half	b	b	b	< 1
	back half				6.6
Hexachlorobutadiene	front half	a	a	a	< 1
	back half				< 0.5
		Output rate (g/min)			
Hexachlorocyclopentadiene	front half	< 0.0001	< 0.0001	< 0.0001	< 0.0002
	back half	< 0.0001	< 0.0001	< 0.0001	< 0.0001
	total	< 0.0002	< 0.0002	< 0.0002	< 0.0003
Bis-(2-ethyl hexyl)-phthalate	front half	0.00030	0.00020	0.00038	0.00025
	back half	0.00036	0.00040	0.00032	0.00034
	total	0.00066	0.00060	0.00070	0.00059
Chlordane	front half	< 0.0001	< 0.0001	< 0.0001	a
	back half	< 0.0001	< 0.0001	< 0.0001	
	total	< 0.0002	< 0.0002	< 0.0002	
Naphthalene	front half	b	b	b	< 0.0002
	back half				0.0011
	total				0.0011
Hexachlorobutadiene	front half	a	a	a	< 0.0002
	back half				< 0.0001
	total				< 0.0003

^a This compound was not detected in the feed during this run.

^b Naphthalene was detected as a PIC in Runs 1 through 3.

TABLE J-10. CONCENTRATION OF VOLATILE PICs IN STACK EFFLUENT
(NOT BLANK CORRECTED), ng/L

		Run 1									
		Fast VOST						Slow VOST			
Gas bag ^a		1st pair	3rd pair	5th pair	6th pair	Avg.		1st pair	2nd ^c pair	Avg.	
Bromochloromethane	9.0	2.1	0.2	0.2	< 0.1	0.6		5.8	14	-	
Bromodichloromethane	0.7	0.7	0.1	0.1	< 0.2	0.2		0.9	4.8	-	
Dibromochloromethane	0.3	< 0.2	< 0.2	0.02	< 0.2	-		0.6	1.2	-	
Bromoform	1.3	< 0.2	< 0.2	0.3	1.5	-		3.3	1.7	-	

		Run 2									
		Fast VOST						Slow VOST			
Gas bag	Gas bag ^a	1st pair	3rd pair	5th pair	6th pair	Avg.		1st pair	2nd pair	Avg.	
Bromochloromethane	< 0.5	5.3	6.8	4.4	7.0	6.0		7.9	9.8	8.8	
Bromodichloromethane	< 1.0	5.2	2.3	3.9	4.8	4.0		7.5	15	11	
Dibromochloromethane	< 1.0	1.8	2.0	2.4	1.7	2.0		9.8	5.7	7.8	
Bromoform	< 1.0	4.4	7.8	37	6.1	14		42	20	31	

(continued)

TABLE J-10. (continued)

	Run 3					
	Fast VOST			Slow VOST		
	Gas bag ^a	1st pair	3rd pair	5th pair	6th pair	Avg.
Bromochloromethane	< 0.3	5.4	9.0	6.4	6.0	6.9
Bromodichloromethane	< 0.7	8.4	9.5	8.4	5.5	8.0
Dibromochloromethane	< 0.7	5.9	18 ^f	3.8	2.4	7.5
Bromoform	1.5	10	240 ^f	18	12	70
						56

	Run 4 ^e					
	Fast VOST			Slow VOST		
	Gas bag	2nd pair	4th pair	5th pair	6th pair	Avg.
Bromochloromethane	12	6.8	6.1	6.4	6.6	6.5
Bromodichloromethane	6.3	2.1	3.9	2.3	0.3	2.1
Dibromochloromethane	7.0	1.3	2.6	2.1	2.6	2.2
Bromoform	53	3.0	11	11	13	9.3

^a This sample was a grab sample.

^b Sample saturated mass spectrometer.

^c The back trap of this pair of traps was not evaluated. These numbers are based on the front trap only.

^d The first and third pairs were not evaluated, the second and fourth pairs were used in their place.

^e Only fast VOST was used in Run 4.

^f The quantification of these numbers was based on a minor ion in the GC/MS, and therefore they are not as precise as the other numbers presented.

TABLE 3-11. EMISSION RATES OF VOLATILE PICs IN STACK EFFLUENT
(NOT BLANK CORRECTED), g/min

	Gas bag ^a	Run 1						
		Fast			VOST			
		1st pair	3rd pair	5th pair	6th pair	Avg.	1st pair	2nd pair ^b
Bromochloromethane	0.00097	0.00022	0.000017	0.000017	< 0.000011	0.000065	0.00063	0.0015
Bromodichloromethane	0.000073	0.00080	0.000012	0.000013	< 0.00002	0.000026	0.000095	0.00052
Dibromochloromethane	0.000037	< 0.00002	< 0.00002	0.0000023	< 0.00002	-	0.000064	0.00013
Bromoform	0.00014	< 0.00002	< 0.00002	0.000036	0.00016	-	0.00036	0.00018

	Gas bag	Run 2						
		Fast			VOST			
		1st pair	3rd pair	5th pair	6th pair	Avg.	1st pair	2nd pair
Bromochloromethane	< 0.00007	0.00030	0.00078	0.00097	0.00063	0.00099	0.00011	0.0014
Bromodichloromethane	< 0.0001	0.00039	0.00074	0.00033	0.00056	0.00069	0.0011	0.0022
Dibromochloromethane	< 0.0001	0.000093	0.00026	0.00028	0.00034	0.00024	0.0014	0.00082
Bromoform	< 0.0001	0.00054	0.00063	0.0011	0.0053	0.00087	0.0060	0.0028
						0.0020		0.0044

TABLE J-11. (continued)

		Run 3					
		Fast VOST			Slow VOST		
Gas bag		1st pair	3rd pair	5th pair	6th pair	1st pair	2nd pair
Bromochloromethane	< 0.00005	0.00077	0.0013	0.00092	0.00087	0.00074	0.00096
Bromodichloromethane	< 0.0001	0.0012	0.0014	0.0012	0.00080	0.0016	0.00092
Dibromochloromethane	< 0.0001	0.00085	0.0025	0.00055	0.00035	0.0015	0.00057
Bromoform	0.00022	0.0014	0.035	0.0027	0.0017	0.0057	0.010
					0.010		0.008
		Run 4 ^c					
		Fast VOST ^c			Avg.		
Gas bag		2nd pair	4th pair	5th pair	6th pair		
Bromochloromethane	0.0020	0.0012	0.0010	0.0011	0.0011	0.0011	
Bromodichloromethane	0.0011	0.00036	0.00066	0.00040	0.00048	0.00059	
Dibromochloromethane	0.0012	0.00021	0.00044	0.00035	0.00044	0.00037	
Bromoform	0.0090	0.00050	0.0019	0.0018	0.0023	0.0016	

^a This sample was a grab sample.

^b This sample based on only the first trap of the pair of traps.

^c No slow VOST in Run 4.

^d This sample saturated the mass spectrometer.

^e The first and third pairs were not analyzed. The second and fourth pairs were analyzed in their place.

^f The quantification of these numbers was based on a minor ion in the GC/MS, and therefore they are not as precise as the other numbers presented.

TABLE J-12. EMISSIONS OF SEMIVOLATILE PICs (NOT BLANK CORRECTED)

Compound		Emission concentration (ng/L)			
		Run 1	Run 2	Run 3	Run 4
Naphthalene	front half	0.053	< 1	< 1	a
	back half	32	12	4.0	
		Output rate (g/min)			
Naphthalene	front half	0.000006	< 0.0001	< 0.0001	a
	back half	0.0035	0.0017	0.00058	
	total	0.0035	0.0017	0.00058	

^a Naphthalene was found in concentrations greater than 100 µg/g in the waste feed in Run 4. It was treated as a POHC.

BLANK VALUES FOR POHCs AND PICs

The average blank value and the standard deviation of the blank population are presented for volatile POHCs and PICs in Table J-13. The sample volumes for gas bags as well as slow and fast VOST are presented in Table J-14. Blank correction data for semivolatile POHCs and PICs are presented in Table J-15. Sample volumes for the MM5 train can be found in Table J-17.

DREs FOR VOLATILES AND SEMIVOLATILES

Destruction and removal efficiencies (DREs) based on the above data are presented in Table J-16. These values were calculated using blank corrected emission rates. Blank corrections involve subtracting the average blank value from the sample value whenever the sample value exceeds the blank by at least three standard deviations of the blank population. The DRE is then calculated using the following formula:

$$\text{DRE} = \frac{\text{input (g/min)} - \text{blank corrected emission rate (g/min)}}{\text{input (g/min)}} \times 100$$

PARTICULATE EMISSIONS

Particulate emissions were measured using a Modified Method 5 train. The data associated with the MM5 train are presented in Table J-17. Particulate emission data are presented in Table J-18. The average particulate concentration (corrected to 7% O₂) for the four runs was 168 mg/dscm (0.074 gr/dscf), compared to the EPA regulatory maximum of 180 mg/dscm (0.08 gr/dscf). The total particulate emission rate averaged 0.69 kg/hr (1.51 lb/hr) for the four runs.

CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

Table J-19 summarizes chloride input and output rates. The data show an average output of 0.16 kg/hr (0.35 lb/hr); below the EPA maximum of 1.8 kg/hr. The average removal efficiency for the four runs was 98.2%. The pH values for the inflow and outflow streams are also presented in Table J-19.

ANALYSIS OF WASTE FEEDS

A general analysis was performed on the waste feeds to determine bromine, chlorine, water, and ash content, as well as heating value and viscosity. An ultimate analysis was performed on the organic and solid waste samples. The results are presented in Table J-20.

CONTINUOUS MONITORING DATA

Continuous monitors were used to determine concentrations of O₂, CO₂, CO, and HC in the stack effluent throughout the four test periods. The results are summarized in Table J-21.

TABLE J-13. VOLATILE BLANK CORRECTION VALUES

	Gas bags		VOST	
	Average blank value (ng)	Standard deviation (ng)	Average blank value (ng)	Standard deviation (ng)
<u>POHCs</u>				
Methylene chloride	90	66	29	11
Chloroform	7.0	6.8	7.8	6.2
Methylene bromide	< 1	0	< 1	0
1,1,1-Trichloroethane	34	52	2.7	4.4
Carbon tetrachloride	1.5	2.2	4	9.8
Trichloroethylene	92	65	7.3	14
Benzene	15	4.9	28	15
Tetrachloroethylene	6.6	4.6	0.27	0.43
Toluene	46	35	12	13
Chlorobenzene	2.3	2.7	0.62	1.5
<u>PICs</u>				
Bromochloromethane	< 1	0	< 1	0
Bromodichloromethane	< 2	0	< 2	0
Dibromochloromethane	< 2	0	< 2	0
Bromoform	< 2	0	7.3	16

TABLE J-14. VOLATILE SAMPLE VOLUMES
(DRY STANDARD LITERS)

Gas bags		Run No.	VOST			
			Fast		Slow	
Run No.	Volume (L)	Run No.	Pair No.	Volume (L)	Pair No.	Volume (L)
1 ^a	5.0	1	1	20.2		
2	2.0	1	3	19.4	1	11.2
2 ^a	2.0	1	5	18.8		
3	3.0	1	6	19.1	2	11.4
4	3.0	2	1	18.4		
		2	3	19.1	1	12.2
		2	5	18.8		
		2	6	18.7	2	11.2
		3	1	18.1		
		3	3	19.9	1	11.8
		3	5	20.3		
		3	6	20.1	2	11.8
		4	2	20.0		
		4	4	19.6		
		4	5	18.8		
		4	6	19.7		

^a This gas bag was a grab sample.

TABLE J-15. SEMIVOLATILE BLANK CORRECTION VALUES

Compound	Blank correction value (µg)
<u>POHCs</u>	
Hexachlorocyclopentadiene	< 1
Bis(2-ethylhexyl)phthalate	4.6
Chlordane	< 1
Naphthalene	< 2
Hexachlorobutadiene	< 1
<u>PICs</u>	
Naphthalene	< 2

TABLE J-16. DESTRUCTION AND REMOVAL EFFICIENCIES (DREs) FOR THE TWI INCINERATOR (BLANK CORRECTED)

Volatile compounds selected for test	Gas bags				Fast VOST				Slow VOST			
	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4
Methylene bromide	99.9954	> 99.9995	99.9949	99.968	> 99.9987	99.9956	99.964	99.982	99.9941	99.9948		99.975
Carbon tetrachloride	99.99946	> 99.9995	> 99.99930	> 99.9988	> 99.9984	> 99.9983	> 99.9987	> 99.99903	99.9963	99.9984		99.9988
Trichloroethylene	> 99.992	> 99.985	> 99.9932	> 99.9937	> 99.9962	99.9945	99.9917	> 99.9989	99.9930	99.9918		99.9978
Benzene	99.9963	> 99.99924	99.9966	99.982	99.9983	99.989	99.984	99.988	99.9963	99.9918		99.9911
Toluene	99.9977	> 99.99975	99.99912	99.985	99.99946	99.9908	99.9963	99.9922	99.9986	99.9964		< 99.998
Volatile compounds occurring in low levels												
Methylene chloride	99.48	> 99.74	> 99.88	> 99.05	> 99.918	99.71	99.88	99.63	> 99.30	99.930		99.87
Chloroform	97.8	> 99.84	> 99.68	99.49	> 99.944	98.2	97.8	99.78	98.0	97.4		97.4
1,1,1-Trichloroethane	> 99.75	> 99.951	> 99.943	> 99.51	99.966	99.81	99.86	99.82	99.80	99.72		99.82
Tetrachloroethylene	99.74	> 99.963	> 99.930	> 99.936	99.965	99.78	99.88	99.982	99.79	99.74		99.88
Chlorobenzene	99.46	> 99.9928	> 99.986	> 99.90	99.965	99.70	99.956	99.966	99.65	99.74		99.940
Semivolatile compounds selected for test												
HPL												
Hexachlorocyclopentadiene	> 99.99	> 99.99	> 99.99	> 99.99	> 99.9996							
Chlordane	> 99.9998	> 99.9999	> 99.9999	> 99.9999	c							
Semivolatile compounds occurring in low levels												
Bis (2-ethyl hexyl) phthalate	99.951		99.960	99.940	99.88							
Naphthalene	c		c	c	99.996							
Hexachlorobutadiene	c		c	c	> 99.98							

a Compounds selected for the test; greater than 1% concentration in a waste feed.

b Compounds occurring at low concentrations; less than 1% in all waste feeds.

c Waste feed concentration < 100 µg/g in this run.

TABLE J-17. MODIFIED METHOD 5 TEST DATA

	Run 1	Run 2	Run 3	Run 4
Vol. of gas sampled, Nm ³ (dscf)	1.512 (53.38)	1.976 (69.79)	1.988 (70.20)	2.290 (80.89)
Sampling time (min)	160	160	160	160
Percent isokinetic	101.2	100.0	99.6	98.1
Moisture content (%)	27.0	19.5	21.2	13.3
Percent O ₂ (dry)	14.0	13.9	13.2	15.5
Percent CO ₂ (dry)	5.1	5.5	5.6	4.1
Stack temperature, °C (°F)	66 (151)	59 (139)	62 (143)	52 (125)
Stack height, m (ft)	16 (52)	16 (52)	16 (52)	16 (52)
Stack velocity, m/min (ft/min)	876 (2,874)	1,014 (3,328)	1,035 (3,394)	1,071 (3,513)
Stack flow rate act. m ³ /min (act. ft ³ /min)	178 (6,270)	206 (7,260)	210 (7,400)	217 (7,670)
Stack flow rate Nm ³ /min (dscf/min)	108 (3,810)	143 (5,040)	144 (5,100)	169 (5,960)

TABLE J-18. PARTICULATE CONCENTRATION AND EMISSION RATE DATA

	Particulate concentration			
	Run 1 ^b	Run 2 ^b	Run 3 ^b	Run 4 ^c
<u>Measured</u>				
mg/dscm	86.1	147	61.2	39.7
(gr/dscf)	(0.0376)	(0.0641)	(0.0267)	(0.0173)
<u>Corrected^a</u>				
mg/dscm	172	290	110	102
(gr/dscf)	(0.0751)	(0.1270)	(0.0479)	(0.0443)
Particulate emissions				
<u>Emission Rate</u>				
kg/hr	0.56	1.26	0.53	0.40
(lb/hr)	(1.23)	(2.77)	(1.17)	(0.88)

^a Measured emissions were corrected to 7% O₂ as follows

$$C_{\text{corr}} = \frac{(14\%)}{(21\% - \%O_2)} (C_{\text{measured}})$$

^b Solid waste feed composed of sand and gravel mixed with chlordane.

^c Solid waste feed was high Btu solids containing 5% C-56.

TABLE J-19. CHLORIDE EMISSIONS AND REMOVAL EFFICIENCY

	Run 1	Run 2	Run 3	Run 4
<u>Chloride emissions</u> kg/hr	0.135	0.161	0.251	0.0978
(lb/hr)	(0.298)	(0.355)	(0.553)	(0.216)
<u>Chloride in feed</u> kg/hr	7.8	10.2	7.8	15.6
(lb/hr)	(17.2)	(22.5)	(17.2)	(34.4)
Removal efficiency	98.3%	98.4%	96.8%	99.4%
(based on chloride in feed)				
pH of inflow water	9.6	9.7	9.7	11.7
pH of outflow water	9.4	9.2	9.3	9.8

TABLE J-20. ANALYSIS OF WASTE FEEDS

Waste	Run	Viscosity sus ^a	Percent Br	Percent Cl	Percent H ₂ O	Percent ash	Heating value	
							kJ/kg	(Btu/lb)
Liquid organic	1	82.1		1.67	4.89	3.12	35,063	(15,087)
	2	92.1		1.69	4.70	3.51	34,703	(14,932)
	3	131.2		1.85	5.34	3.86	34,150	(14,694)
	4	116.2		4.92	5.55	3.96	32,170	(13,842)
Aqueous	1		0.0052	0.0097	93.80	< 0.05	158	(68)
	2		< 0.05	0.013	93.60	< 0.05	1,215	(523)
	3		0.10	0.0096	95.70	< 0.05	1,917	(825)
	4		0.014	0.013	95.40	< 0.05	1,917	(825)
Solid	1			1.98	1.50	89.62	811	(349)
	2			2.56	1.38	90.43	790	(340)
	3			1.96	1.60	90.22	672	(289)
	4			5.17	11.13	29.03	20,038	(8,622)
Fuel oil						< 0.05	45,020	(19,371)
Ultimate analysis	Run	Percent C	Percent H	Percent N	Percent S	Percent Cl	Percent ash	Percent O (by difference)
Liquid organic	2	74.23	8.38	0.65	0.14	1.69	3.51	11.40
Solid	4	48.68	6.02	0.018	0.056	5.17	29.03	11.03

^a Saybolt universal seconds.

^b Runs 6 and 7 used the same analysis values as Run 3 for liquid organic and aqueous waste streams since these runs were on the same day.

^c Runs 8A and 8B used the same analysis values as Run 4 for liquid organic and aqueous waste streams since these runs were on the same day.

TABLE J-21. CONTINUOUS MONITORING DATA^a

	Run 1	Run 2	Run 3	Run 4
Oxygen (%)				
Range	11.5 - 13.0	11.3 - 15.6	11.6 - 16.3	14.0 - 16.4
Average	12.4	13.0	13.2	15.6
Carbon dioxide (%)				
Range	5.9 - 7.4	4.3 - 7.5	4.8 - 7.4	3.1 - 4.9
Average	6.6	6.2	6.1	3.9
Carbon monoxide (ppm)				
Range	1.2 - 66.3	< 1 - 13.8	< 1 - 2.3	< 1 - 2.0
Average	4.3	0.9	1.2	< 1
Total hydrocarbon (ppm) ^b				
Range	2.0 - 2.9	1.7 - 2.1	1.3 - 2.2	< 1 - 2.1
Average	2.5	1.9	1.7	< 1

^a Concentrations on dry gas basis.

^b Total hydrocarbon as propane.

POHCs AND PICs IN WATER STREAMS

The streams entering and exiting the quench and scrubber system, as well as the makeup water from city and well sources, were analyzed for volatile and semivolatile POHCs and PICs. The results are presented in Table J-22. The volatiles, chloroform, dibromochloromethane and dichlorobromomethane appear in higher concentrations in the makeup water than in the outflow water. The data suggest that these compounds are being stripped from the makeup waters into the stack effluent gas. Using the flow rates presented in Figure J-1 this stripping could account for nearly all of the emissions of these three compounds as reported earlier in Tables J-8 and J-11.

POHCs IN CHAMBER ASH

Chamber ash samples from each run were analyzed for volatile and semivolatile POHCs. The results are presented in Table J-23. Benzene, toluene, and chlordane were detected in the ash during all four runs. Chlordane was found in the largest quantities.

METALS

Waste feed samples were analyzed for metals content and the results are presented in Table J-24. Barium, chromium, and lead were the most frequently detected metals, with lead appearing in the highest concentrations.

The chamber ash and water tank solids were analyzed for metals content and the results appear in Table J-25. The metal concentrations and emission rates in the particulate are presented in Table J-26. The particulate contained approximately 10% lead and 4% cadmium by weight. The output rate of metals in the particulate is compared with the input rate of metals in the waste feed in Table J-27.

EP TOXICITY TEST

Samples of the combustion chamber ash were analyzed according to the EP toxicity method. The results are presented in Table J-28. None of the concentrations exceeded the maximum limit for EP toxicity.

QUALITY ASSURANCE SUMMARY

Quality assurance (QA) activities were divided into three categories: (1) external auditing; (2) sampling QA activities; and (3) analysis QA activities. The three areas of QA activities were conducted independently of one another but were coordinated to ensure data quality and integrity. The purpose of this section is to summarize QA activities and findings. Detailed QA activities and findings are given in Appendix K.

TABLE J-22. CONCENTRATION OF VOLATILE PORCS AND PICs IN WATER SAMPLES

	Makeup waters (µg/L)								Scrubber waters (µg/L)								Water tank solids (µg/g)
	Well water				City water				Recirculated water (Inlet to quench and scrubber)				Return water (Outlet from quench and scrubber)				
	Run 1 ^a	Run 2 ^a	Run 1 ^b	Run 2 ^b	Run 3 ^c	Run 4 ^c	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4			
PORCS																	
Methylene chloride	20	11	15	2.9	5.4	1.6	4.5	5.8	5.7	2.0	11	6.2	10	< 1	< 1	< 1	
Chloroform	100	65	66	64	71	87	1.6	2.0	3.1	2.6	< 1	< 1	< 1	< 1	< 1	< 1	
Methylene bromide	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
1,1,1-Trichloroethane	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Carbon tetrachloride	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Trichloroethylene	14	9.0	< 1	< 1	< 1.3	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Benzene	> 200 ^e	15	1.1	< 1	< 1	< 1	2.0	< 1	< 1	< 1	< 1	1.6	< 1	< 1	< 1	1.1	
Tetrachloroethylene	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Toluene	140	84	4.6	< 1	< 1	< 1	23	4.9	3.7	1.0	1.9	2.0	2.0	< 1	< 1	3.5	
Chlorobenzene	150	73	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
PICs																	
Bromochloromethane	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Bromodichloromethane	< 1	< 1	16	15	21	25	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Dibromochloromethane	< 1	< 1	19	18	2.5	4.0	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1	
Bromoform	< 1	< 1	< 1	< 1	< 1	< 1	< 1	2.2	4.3	1.1	< 1	2.6	19	1.3	< 1	< 1	

^a Well water used as makeup for demisters in Runs 1 and 2 (approximate flow rate ~ 7 L/min).^b City water used as makeup for recirculation tank in Runs 1 and 2 (approximate flow rate ~ 70 L/min).^c City water was used for all makeup purposes in Runs 3 and 4 with the same flow rates as in Runs 1 and 2.^d Tank solids were also analyzed for semivolatile PORCS. All semivolatile PORC concentrations were < 1 µg/g except chlordane (1.5 µg/g) and bis-(2-ethyl hexyl)-phthalate (4.5 µg/g).^e Compound saturated mass spectrometer.

TABLE J-23. VOLATILE AND SEMIVOLATILE POHCs
IN CHAMBER ASH ($\mu\text{g/g}$)

	Kiln ash			
	Run 1	Run 2	Run 3	Run 4
<u>Volatiles</u>				
Methylene chloride	1.6	< 1	< 1	< 1
Chloroform	< 1	< 1	< 1	< 1
Methylene bromide	2.5	< 1	< 1	< 1
1,1,1-Trichloroethane	< 1	< 1	< 1	< 1
Carbon tetrachloride	< 1	< 1	< 1	< 1
Trichloroethylene	< 1	< 1	< 1	< 1
Benzene	1.0	1.0	1.1	3.9
Tetrachloroethylene	< 1	< 1	< 1	< 1
Toluene	7.9	6.8	4.7	35
Chlorobenzene	< 1	< 1	< 1	< 1
<u>Semivolatiles</u>				
Hexachlorocyclo- pentadiene	1.6	< 1	< 1	< 1
Bis-(2-ethyl hexyl)- phthalate	< 1	< 1	< 1	< 1
Naphthalene	< 1	< 1	< 1	41
Chlordane	570	170	59	17
Hexachlorobutadiene	< 1	< 1	< 1	< 1

TABLE J-24. METAL CONCENTRATION IN WASTE FEED SAMPLES (µg/g)

Metal	Liquid organic waste				Solid waste			
	Run 1	Run 2	Run 3	Run 4	Run 1	Run 2	Run 3	Run 4
Ag	< 1	< 1	< 1	< 1	< 1	< 1	< 1	< 1
As	< 23	< 23	< 23	< 23	< 23	< 23	< 23	< 23
Ba	910	990	1,100	1,200	25	37	150	16
Be	< 0.3	< 0.3	< 0.3	< 0.3	0.8	1.3	1.2	1.1
Cd	44	49	55	64	< 2	< 2	< 2	< 2
Cr	230	250	290	320	8.3	14	20	11
Hg	< 50	< 50	< 50	< 50	< 50	< 50	< 50	< 50
Ni	< 4	< 4	< 4	< 4	9	14	18	7
Pb	1,100	1,200	1,300	1,500	< 23	< 23	< 23	47
Sb	< 24	< 24	< 24	< 24	< 24	< 24	< 24	< 24
Se	< 150	< 160	< 160	< 160	< 170	< 290	< 200	< 90
Tl	< 22	< 22	< 22	< 22	< 22	< 22	< 22	< 22

TABLE J-25. METAL CONCENTRATION IN CHAMBER ASH AND
WATER TANK SOLIDS ($\mu\text{g/g}$)

Metal	Chamber ash				Water tank solids
	Run 1	Run 2	Run 3	Run 4	
Ag	1.4	1.1	1.2	1.3	< 14
As	< 23	< 23	< 23	< 23	< 240
Ba	190	280	210	310	4,200
Be	1.0	2.0	2.1	2.6	< 3
Cd	2.1	2.2	< 2.0	2.1	210
Cr	70	91	66	180	730
Hg	< 50	< 50	< 50	< 50	< 290
Ni	12	20	11	24	< 35
Pb	81	110	94	79	8,000
Sb	25	47	31	28	< 230
Se	< 200	< 390	< 210	< 400	< 2,800
Tl	< 22	< 22	< 22	< 22	< 190

TABLE J-26. METAL EMISSIONS IN STACK PARTICULATE

Metal	Concentration in particulate ($\mu\text{g/g}$)			
	Run 1	Run 2	Run 3	Run 4
Ag	< 540	< 240	< 580	< 770
As	< 2,000	900	< 2,200	< 2,800
Ba	770	41	580	480
Be	< 12	6.2	< 12	< 16
Cd	4,000	3,800	4,300	3,700
Cr	1,500	830	1,000	1,000
Hg	< 2,200	< 960	< 2,300	< 3,100
Ni	< 460	230	< 500	< 660
Pb	95,000	96,000	100,000	100,000
Sb	< 6,200	< 2,800	< 6,700	< 8,800
Se	< 49,000	< 22,000	< 53,000	< 70,000
Tl	< 4,000	< 1,800	< 4,300	< 5,700
Metal	Emission rate (g/min)			
	Run 1	Run 2	Run 3	Run 4
Ag	< 0.005	< 0.005	< 0.005	< 0.005
As	< 0.02	0.019	< 0.02	< 0.02
Ba	0.0072	0.0087	0.0051	0.0032
Be	< 0.0001	0.00013	< 0.0001	< 0.0001
Cd	0.036	0.080	0.036	0.025
Cr	0.014	0.017	0.0087	0.0068
Hg	< 0.02	< 0.02	< 0.02	< 0.02
Ni	< 0.004	< 0.004	< 0.004	< 0.004
Pb	0.88	2.0	0.87	0.68
Sb	< 0.06	< 0.06	< 0.06	< 0.06
Se	< 0.5	< 0.5	< 0.5	< 0.5
Tl	< 0.04	< 0.04	< 0.04	< 0.04

TABLE J-27. METAL IN STACK EMISSIONS AS PERCENT OF INPUT
IN WASTE FEEDS^a

Metal	Run 1	Run 2	Run 3	Run 4
Ag	b	b	b	b
As	b	b	b	< 20
Ba	0.22	0.24	0.14	0.22
Be	< 3	2.2	< 2	< 2
Cd	24	47	20	34
Cr	0.84	1.8	0.89	1.6
Hg	b	b	b	b
Ni	< 10	< 6	< 6	< 10
Pb	24	48	21	36
Sb	b	b	b	b
Se	b	b	b	b
Tl	b	b	b	b

^a Percent = $\frac{\text{stack output, g/min}}{\text{waste input, g/min}} \times 100$

^b Concentration in waste input was below detection limits.

TABLE J-28. RESULTS OF EP TOXICITY TEST FOR CHAMBER ASH

	Concentration in ash sample leachate ^a (mg/L)				Maximum concentration limit in leachate ^b for EP toxicity ^b (mg/L)
	Run 1	Run 2	Run 3	Run 4	
<u>Metals</u>					
Silver	< 0.009	< 0.009	< 0.009	< 0.009	5.0
Arsenic	0.033	0.016	< 0.001	0.015	5.0
Barium	0.36	0.34	0.46	0.80	100.0
Cadmium	0.016	0.023	0.0088	0.0084	1.0
Chromium	0.014	0.013	< 0.007	0.015	5.0
Lead	0.12	0.10	0.058	< 0.04	5.0
Mercury	< 0.001	< 0.001	< 0.001	< 0.001	0.2
Selenium	< 0.010	< 0.010	0.13	< 0.010	1.0
<u>Organics</u>					
Endrin	0.007	0.003	< 0.002	< 0.002	0.02
Lindane	< 0.001	< 0.001	< 0.001	< 0.001	0.4
Methoxychlor	< 0.005	< 0.005	< 0.005	< 0.005	10.0
Toxaphene	< 0.05	< 0.05	< 0.05	< 0.05	0.5
2,4-D	< 0.2	< 0.2	< 0.2	< 0.2	10.0
2,4,5-TP	< 0.02	< 0.02	< 0.02	< 0.02	1.0

^a Ash samples were leached according to EP toxicity procedures in Federal Register, May 19, 1980.

^b Federal Register, May 19, 1980, p. 33122.

External Audits

This area of QA activity was conducted under the direction of the Program QA Monitor, Dr. D. Takade, and by QA personnel. Performance audits (quantitative evaluations) and systems audits (qualitative evaluations) were conducted for the analysis phases of the project.

A performance audit and limited systems audit were conducted for the laboratory analysis phase of the project. Performance audit samples were prepared and analyzed prior to a particular analysis. Performance audits were conducted on GC/MS analysis of volatile organics and atomic absorption analysis of arsenic.

Results of the audit demonstrated adequate and acceptable performance for GC/MS analysis of volatile organics; however, some problems were encountered with arsenic analysis. This portion of the analytical program is judged to be within control limits.

Systems audits demonstrated that laboratory analyses were done according to protocol.

Sampling QA Activities

In general, sampling QA activities are scrutinized as follows. All equipment tests, calibration, and maintenance programs are specified prior to any field sampling activities, and field sampling equipment is tested and, if necessary, recalibrated upon return from the test. Equipment shop records indicate that this function proceeded in an acceptable manner.

Analysis QA Activities

Several internal quality control procedures were specifically designed for this program to ensure that the sample preparations and analyses were performed properly and that the instruments were operating according to predetermined acceptance criteria. Samples to be extracted were first fortified with surrogate compounds whose recoveries were monitored during sample analysis. If the recoveries were outside a certain acceptance range, this suggested a problem with the sample workup or analysis. Other internal quality control procedures excluded spiking all samples, just prior to analysis, with internal standards. The internal standards were used to quantify the sample components and to monitor the GC/MS reproducibility and reliability. Another internal quality control procedure was to monitor the ion abundance of reference compounds spiked into the samples, which indicated how well the mass spectrometers were providing mass spectra whose ion abundances were comparable with those in the mass spectral reference libraries.

SECTION 6

RESULTS FOR EXPERIMENTAL TEST RUNS 6, 7, 8A, AND 8B AT THE TWI INCINERATOR

In addition to the four test runs discussed in Sections 1 through 5, four short runs (20-min each) were conducted to test the effects of altered conditions in the incinerator on emissions. This section summarizes the differences in operating conditions and sampling and analysis methods between these short runs and the four test runs presented earlier. Results for Runs 6, 7, 8A, and 8B are also presented.

OPERATING CONDITIONS

Process data and waste feed data for Runs 6, 7, 8A, and 8B are presented in Tables J-29 and J-30, respectively. Runs 6 and 7 were conducted following Run 3, on the same day. No solid waste was fed to the chamber during these two runs. The liquid organic waste feed rate was increased in order to increase the heat input to the combustion chamber. There also was an attempt to change the distribution of combustion air in the chambers. These changes were made to try and increase the levels of CO and THC emitted from the stack.

Runs 8A and 8B were conducted following Run 4, on the same day. As in Runs 6 and 7 the liquid organic feed rate was increased and the combustion air altered in an attempt to raise CO and THC levels. In addition, at the beginning of each run, a 20-lb batch of solid waste was fed to the chamber. This waste was a high Btu sludge known from previous experience to be accompanied by an increase in CO levels.

SAMPLING AND ANALYSIS

The sampling for these four short runs could not be as extensive as in Runs 1 through 4. Although the methods for sampling and analysis were the same, they were abbreviated due to the short sampling time (20 min) and no MM5 sampling could be done for particulate, HCl, or semivolatile POHCs. The waste feed was analyzed for volatile POHCs and the stack gases were sampled for volatile POHC and PIC emissions. Gas bags were used on Runs 6 and 7, but not in Runs 8A and 8B. The VOST train was also operated for the duration of each of the four runs (i.e., 20 min, or one pair of VOST traps). It was operated at the flow rate used for slow VOST sampling in the earlier runs (approximately 18 liters/hr). Also in Runs 8A and 8B gas bulb grab samples were taken for GC/ECD analysis and these results are presented in Section 7.

TABLE J-29. PROCESS DATA (FROM PLANT INSTRUMENTS)

	Run 6 ^a	Run 7 ^a	Run 8A ^{a,b}	Run 8B ^{a,b}
Waste feed heat load, ^c 10 ⁶ kJ/hr (10 ⁶ Btu/hr)	9.5 (9.0)	11.5 (10.9)	9.3 (8.8)	10.5 (9.9)
Primary chamber temp., °C ^d (°F)	1,220 (2,230)	1,110 (2,020)	1,120 (2,050)	1,120 (2,040)
Secondary chamber temp., °C ^d (°F)	1,150 (2,110)	1,120 (2,050)	1,160 (2,120)	1,170 (2,140)
Gas after prequench temp., °C (°F)	82 (180)	84 (183)	79 (175)	82 (180)
O ₂ , %	13.9	13.8	14.5	12.5
DP across venturi, kPa (in.wc)	2.0 (8)	2.0 (8)	2.0 (8)	2.0 (8)
H ₂ O flow to venturi, L/min (gpm)	190 (51)	190 (51)	200 (54)	200 (54)
Organic waste feed rate, L/min (gpm)	3.3 (0.88)	3.8 (1.0)	3.9 (1.0)	4.2 (1.1)
Aqueous waste feed rate, L/min (gpm)	2.8 (0.75)	2.8 (0.75)	3.2 (0.86)	1.9 (0.50)
Organic waste atomizing air pressure, kPa (psig)	69 (10)	110 (16)	41 (6)	41 (6)
Organic waste feed pressure, kPa (psig)	33 (4.8)	43 (6.2)	54 (7.8)	62 (9.0)
Aqueous waste feed pressure, kPa (psig)	69 (10)	63 (9.2)	30 (4.3)	36 (5.2)
Combustion chamber residence time, sec	3.0	3.0	2.8	2.8
Stack height, m (ft)	16 (52)	16 (52)	16 (52)	16 (52)
Stack velocity, m/min (ft/min)	818 (2,680)	823 (2,700)	887 (2,910)	887 (2,910)
Stack temperature, °C (°F)	67 (152)	66 (151)	59 (139)	62 (143)

^a Runs 6, 7, 8A and 8B were 20 min runs.

^b Runs 8A and 8B used a special solid waste feed.

^c Heat load calculations based on HHV of wastes; does not include auxiliary fuel.

^d Temperature as measured by thermocouples that projected into gas stream. Thermocouples even with refractory were not used because it was determined that they had been replaced with wrong type of thermocouple wire.

TABLE J-30. WASTE FEED RATES,^a kg/min (lb/min)

	Run 6 ^b	Run 7 ^b	Run 8A ^c	Run 8B ^c
Aqueous waste	7.6 (16.7)	9.1 (20.0)	4.7 (10.3)	6.4 (14.1)
Liquid organic waste ^f	3.9 (8.6)	4.7 (10.3)	4.1 (9.0)	4.5 (10.0)
Solid waste				
High Btu	d	d	0.45 ^e	0.45 ^e
Ink sludge			(1.0)	(1.0)

^a Flow rates for fuel oil are not known for these four runs.

^b Feed rates for Runs 6 and 7 were determined from the change in waste storage tank levels.

^c Liquid and aqueous feed rates for Runs 8A, and 8B were calculated from flow meter readings, ratioed against flow rates calculated in previous runs.

^d No solids were fed during Runs 6 and 7.

^e One 20-lb batch of solid waste was fed near beginning of 20-min test period.

^f Organic feed rates calculated from volumetric flow rates using an average density of 0.923 kg/L for the waste.

RESULTS

The waste feeds for the four different runs were analyzed for volatile POHCs and the results are presented in Table J-31. POHC input rates are listed in Table J-32 but the input of POHCs in the fuel oil are not included since fuel oil flow rates could not be measured during these short runs.

The results from the gas bag and VOST samples for volatile POHCs in the effluent gas are presented as concentrations in Table J-33 and as emission rates in Table J-34. Volatile PICs are also presented in terms of concentration (Table J-35) and emission rates (Table J-36). As in earlier runs, stripping of some volatile POHCs (e.g., chloroform, dibromochloromethane, and bromodichloromethane) from the scrubber water could have contributed to higher stack concentrations. Blank correction values are found in Section 5, Table J-13 for volatile compounds. Sample volumes are found in Table J-37. DREs for volatile and semivolatile POHCs are listed in Table J-38.

The stack flow data used to calculate emission rates are found in Table J-39. Stack flows for these short runs were based on single point monitoring of stack velocity. Continuous monitoring data for CO₂, O₂, CO, and hydrocarbons are found in Table J-40. The CO concentrations in the stack were somewhat higher in Runs 7, 8A, and 8B than in Runs 1 through 4 as intended. A general analysis was performed on the waste feeds to determine bromine, chlorine, water and ash content, as well as heating value. These results are presented in Table J-41.

TABLE J-31. CONCENTRATIONS OF VOLATILE POHCs IN WASTE FEED - RUNS 6, 7, 8A, AND 8B ($\mu\text{g/g}$)

Compound	Liquid organic waste				Liquid aqueous waste				Solid waste			Fuel oil ^g
	Run 6	Run 7	Run 8A	Run 8B	Run 6 ^a	Run 7 ^a	Run 8A ^b	Run 8B ^b	Run 6 ^c	Run 7 ^c	Run 8A	Run 8B
Methylene chloride	270	200	110	130	60	60	68	68	< 10	< 10	< 10	440
Chloroform	240	140	100	120	< 10	< 10	< 10	< 10	< 10	< 10	< 10	23
Methylene bromide	< 10	190	< 10	< 10	4,700	4,700	5,700	5,700	160	160	160	210
1,1,1-Trichloroethane	470	260	360	310	< 10	< 10	< 10	< 10	31	31	31	220
Carbon tetrachloride	13,000	11,000	13,000	11,000	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Trichloroethylene	12,000	10,000	12,000	10,000	< 10	< 10	< 10	< 10	< 10	< 10	< 10	20
Benzene	74,000 ^d	74,000 ^d	74,000 ^d	74,000 ^d	< 10	< 10	< 10	< 10	20	20	20	91
Tetrachloroethylene	120	110	90	110	< 10	< 10	< 10	< 10	< 10	< 10	< 10	380
Toluene	250,000 ^e	250,000 ^e	250,000 ^e	250,000 ^e	20	20	18	18	> 1,000 ^f	> 1,000 ^f	> 1,000 ^f	1,500
Chlorobenzene	500	450	420	420	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10

^a Aqueous waste for Runs 6 and 7 was assumed to have the same composition as that in Run 3 since these runs were on the same day.

^b Aqueous waste for Runs 8A and 8B was assumed to have the same composition as that in Run 4 since these runs were on the same day.

^c No solid waste was fed in Runs 6 and 7.

^d Benzene saturated the mass spectrometer in all samples except one, which was analyzed specifically to determine benzene concentration. This sample is used as representative for all runs.

^e Toluene saturated or truncated in the mass spectrometer on all runs. This value (250,000 $\mu\text{g/g}$) is based on information on the waste feed obtained from the plant.

^f Compound saturated mass spectrometer, but was not believed to be greater than 20,000 $\mu\text{g/g}$.

^g Only one fuel oil sample was analyzed, and assumed representative for all runs.

TABLE J-32. VOLATILE POHC INPUT RATES^a - RUNS 6, 7, 8A, AND 8B (g/min)

	Run 6	Run 7	Run 8A	Run 8B
Methylene chloride	1.5 ^b	1.5 ^b	0.77 ^b	1.0 ^b
Chloroform	0.94 ^b	0.66 ^b	0.41 ^b	0.54 ^b
Methylene bromide	37	44	27	37
1,1,1-Trichloroethane	1.8 ^b	1.2 ^b	1.5 ^b	1.4 ^b
Carbon tetrachloride	24	52	49	50
Trichloroethylene	110	40	62	63
Benzene	290	350	300	330
Tetrachloroethylene	0.47 ^b	0.52 ^b	c	0.50 ^b
Toluene	980	1,180	1,020	1,120
Chlorobenzene	2.0	2.1	1.7	1.9

^a These input rates do not include any input of POHCs in fuel oil.

^b The presence of POHCs in the fuel oil could significantly increase these input rates.

^c This compound not present in waste feed in concentrations greater than 100 µg/g for this run.

TABLE J-33. CONCENTRATION OF VOLATILE POHCs IN STACK EFFLUENT -
RUNS 6, 7, 8A, AND 8B (NOT BLANK CORRECTED), ng/L

	Gas bags ^a		Slow VOST ^a			
	Run 6	Run 7	Run 6	Run 7	Run 8A	Run 8B
Methylene chloride	46	30	48	47	9.7	5.6
Chloroform ^c	21	0.6	52	40	3.8	2.4
Methylene bromide	13	3.0	57	28	0.4	< 0.3
1,1,1-Trichloroethane	7.0	20	14	15	59	10
Carbon tetrachloride	5.0	1.3	11	4.0	13	14
Trichloroethylene	50	15	74 ^b	19	3.6	2.6
Benzene	160	140	180	110	15	3.8
Tetrachloroethylene	6.5	5.0	10 ^b	6 ^b	1.0	1.0
Toluene	200	90	580 ^b	280	32	9.7
Chlorobenzene	26	46	48	34	3.0	0.5

^a Runs 6, 7, 8A, and 8B were short runs (20 min). Therefore, VOST results are for only one pair of traps for each run.

^b The quantification of these numbers was based on a minor ion in the GC/MS, and therefore, they are not as precise as the other numbers presented.

^c This compound may have been stripped from the scrubber water.

TABLE J-34. EMISSION RATE OF VOLATILE POHCs IN STACK EFFLUENT -
RUNS 6, 7, 8A, AND 8B (NOT BLANK CORRECTED), g/min

	Gas bags ^a		Slow VOST ^a			
	Run 6	Run 7	Run 6	Run 7	Run 8A	Run 8B
Methylene chloride	0.0076	0.0051	0.0079	0.0078	0.0017	0.0010
Chloroform	0.0035	0.000091	0.0086	0.0067	0.00068	0.00044
Methylene bromide	0.0022	0.00049	0.0094	0.0047	0.000068	< 0.00006
1,1,1-Trichloroethane	0.0012	0.0033	0.0022	0.0025	0.011	0.0018
Carbon tetrachloride	0.00083	0.00022	0.0018	0.00067	0.0023	0.0026
Trichloroethylene	0.0083	0.0025	0.012	0.0031	0.00065	0.00047
Benzene	0.026	0.024	0.030 ^b	0.018	0.0027	0.00068
Tetrachloroethylene	0.0011	0.00083	0.0017	0.0010	0.00017	0.00018
Toluene	0.032	0.015	0.095 ^b	0.047 ^b	0.0058	0.0017
Chlorobenzene	0.0044	0.0077	0.0080	0.0057	0.00053	0.000097

^a Runs 6, 7, 8A, and 8B were short runs (20 min). Therefore, VOST results are for only one pair of traps for each run.

^b The quantification of these numbers was based on a minor ion in the GC/MS, and therefore, they are not as precise as the other numbers presented.

^c This compound may have been stripped from the scrubber water.

TABLE J-35. CONCENTRATION OF VOLATILE PICs IN STACK EFFLUENT -
RUNS 6, 7, 8A, AND 8B (NOT BLANK CORRECTED), ng/L

	Gas bags ^a		Slow VOST ^a			
	Run 6	Run 7	Run 6	Run 7	Run 8A	Run 8B
Bromochloromethane ^c	1.4	0.4	1.8	3.2	< 0.3	4.3
Bromodichloromethane ^c	12	< 1	5.9	3.3	< 0.7	< 0.7
Dibromochloromethane	9.5	0.5	7.1	3.2	< 0.7	< 0.7
Bromoform	48	28	240 ^b	240	16	< 0.7

^a Runs 6, 7, 8A, and 8B were short runs (20 min). Therefore, VOST results are for only one pair of traps for each run.

^b The quantification of these numbers was based on a minor ion in the GC/MS, and therefore, they are not as precise as the other numbers presented.

^c These compounds may have been stripped from the scrubber water.

TABLE J-36. EMISSION RATE OF VOLATILE PICs IN STACK EFFLUENT -
RUNS 6, 7, 8A, AND 8B (NOT BLANK CORRECTED), g/min

	Gas bags ^a		Slow VOST ^a			
	Run 6	Run 7	Run 6	Run 7	Run 8A	Run 8B
Bromochloromethane	0.00024	0.000058	0.00029	0.00053	< 0.00006	0.00077
Bromodichloromethane ^c	0.0019	< 0.0002	0.00098	0.00056	< 0.0001	< 0.0001
Dibromochloromethane ^c	0.0016	0.000083	0.0012 ^b	0.00053	< 0.0001	< 0.0001
Bromoform	0.0079	0.0046	0.039 ^b	0.040	0.0028	< 0.0001

^a Runs 6, 7, 8A, and 8B were short runs (20 min). Therefore, VOST results are for only one pair of traps for each run.

^b The quantification of these numbers was based on a minor ion in the GC/MS, and therefore, they are not as precise as the other numbers presented.

^c These compounds may have been stripped from the scrubber water.

TABLE J-37. VOLATILE SAMPLE VOLUMES (DRY
STANDARD LITERS)

Gas bag		Slow VOST	
Run No.	Volume (L)	Run No.	Volume (L)
6	2.0	6	8.0
7	2.0	7	6.0
		8A	6.1
		8B	6.1

TABLE J-38. DESTRUCTION AND REMOVAL EFFICIENCIES (DRES) FOR EXPERIMENTAL RUNS 6, 7, 8A, AND 8B AT THE TWI INCINERATOR (BLANK CORRECTED)

Volatile compounds selected for test ^a	Gas bags		Slow Vost			
	Run 6	Run 7	Run 6	Run 7	Run 8A	Run 8B
Methylene bromide	99.9942	99.9989	99.974	99.9936	99.99981	> 99.99992
Carbon tetrachloride	99.9973	> 99.99958	99.9926	> 99.9987	99.9966	99.9951
Trichloroethylene	> 99.9924	99.9938	99.989	99.9926	> 99.99921	> 99.99924
Benzene	99.9910	99.9932	99.990	99.9950	99.99952	> 99.99979
Toluene	99.9970	99.9990	< 99.9979	< 99.9976	99.99959	99.99988
Volatile compounds occurring in low levels						
Methylene chloride	> 99.50	> 99.66	99.51	99.53	> 99.83	> 99.90
Chloroform	99.69	> 99.986	99.10	99.02	> 99.88	> 99.920
1,1,1-Trichloroethane	> 99.935	> 99.72	99.88	99.85	99.47	99.87
Tetrachloroethylene	> 99.77	> 99.84	99.64	99.81	c	99.966
Chlorobenzene	99.79	99.64	99.60	99.73	99.978	> 99.9949

^a Compounds selected for test; greater than 1% concentration in a waste feed.

^b Compounds occurring at low concentrations; less than 1% in all waste feeds.

^c Waste feed concentration < 100 µg/g in this run.

TABLE J-39. STACK FLOW DATA

	Run 6	Run 7	Run 8A	Run 8B
Stack temperature, °C (°F)	67 (152)	66 (151)	59 (139)	62 (143)
Stack flow rate ^a Nm ³ /min (dscf/min)	166 (5,850)	167 (5,910)	180 (6,360)	180 (6,340)

^a Stack flow rate based on velocity reading at one point in stack.

TABLE J-40. CONTINUOUS MONITORING DATA^a - RUNS 6,
7, 8A, AND 8B

	Runs 6 ^c	Run 7 ^c	Run 8A ^c	Run 8B ^c
Oxygen (%)				
Range	11.9-16.0	11.9-13.0	13.0-17.8	10.4-16.1
Average	13.1	12.4	14.2	13.5
Carbon dioxide (%)				
Range	3.5-6.9	5.9-6.8	2.6-6.7	3.2-8.8
Average	5.9	6.4	5.7	6.7
Carbon monoxide (ppm)				
Range	0-22	9-58	24-150	25-330
Average	2	23	63	120
Total hydrocarbon (ppm) ^b				
Range	1-2	1-2	1-2	2-3
Average	2	2	2	2

^a Concentrations on dry gas basis.

^b Total hydrocarbon as propane.

^c Runs 6, 7, 8A, and 8B were 20-min runs.

TABLE J-41. GENERAL ANALYSIS OF WASTE FEEDS

Waste	Run	Viscosity sus ^a	Percent Br	Percent Cl	Percent H ₂ O	Percent ash	Heating value kJ/kg (Btu/lb)
Liquid organic	6&7 ^b	131.2		1.85	5.34	3.86	34,150 (14,694)
	8A&8B ^c	116.2		4.92	5.55	3.96	32,170 (13,842)
Aqueous	6&7 ^b		0.10	0.0096	95.70	< 0.05	1,917 (825)
	8A&8B ^c		0.014	0.013	95.40	< 0.05	1,917 (825)
Solid	8A&8B			0.42	7.98	12.88	23,175 (9,972)

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^a Saybolt universal seconds.^b Runs 6 and 7 used the same analysis values as Run 3 for liquid organic and aqueous waste streams since these runs were on the same day.^c Runs 8A and 8B used the same analysis values as Run 4 for liquid organic and aqueous waste streams since these runs were on the same day.

SECTION 7

FIELD GC/ECD ANALYSIS OF TWI WASTE FEED AND GAS BULB SAMPLES

In addition to the sampling and analysis methods discussed in Sections 3 and 4 of this appendix, two additional methods were field tested. The first involved the use of a gas chromatograph/electron capture detector (GC/ECD) in the field, to analyze waste feed samples and gas bulb samples for CCl_4 and TCE. The other method involved GC/MS analysis of samples of stack effluent returned to the analytical laboratory in the gas bulbs.

The analysis of samples in the field by GC/ECD was intended for evaluation as a quick screening technique. The method was not optimized prior to or during use in the field. For example, quantification of both gaseous and liquid samples was based on a single point calibration using a liquid calibration standard. Upon return to the analytical laboratory, several of the liquid samples were prepared and analyzed by GC/ECD using procedures similar to those used in the field; however, during the lab analysis there was opportunity to prepare and analyze multipoint calibration standards which bracketed the sample concentration range and to make more replicate injections.

The field and laboratory GC/ECD results did not agree well with each other or with the GC/MS results. The data and analytical methods are discussed in more detail in the sections below.

HISTORICAL

The first GC/ECD analyses of waste feed samples for this program was performed on a set of wastes from two other plants. The samples were diluted in methanol and analyzed on the same type of GC column that has been used to analyze the samples by GC/MS. Using the same GC column has the advantage of providing the same retention order for the compounds and thus the GC/MS data can provide information on coeluting peaks. The waste GC/ECD data agreed fairly well with the GC/MS results. For example, the GC/MS value for CCl_4 in the waste for one plant was 5,200 $\mu\text{g/g}$ and the GC/ECD value was 5,600 $\mu\text{g/g}$. The GC/ECD values for the other plant agreed less well with the GC/MS results. The GC/MS results for CCl_4 in three wastes were 66,000, 68,000, and 72,000 $\mu\text{g/g}$, while the corresponding GC/ECD results were 84,000, 88,000, and 48,000 $\mu\text{g/g}$, respectively.

However, before the GC/ECD method was used in the field at TWI, a different analytical protocol was adopted based on EPA Method 23 for GC analysis of halogenated organics. The GC column used was a 3 m x 3.2 mm I.D. glass column packed with 20% SP-2100/0.1% Carbowax 1500 on 100/120 mesh

Supelcoport. Also, it was decided to use hexane as the solvent for the waste samples rather than methanol, since hexane has no response by GC/ECD.

ANALYSIS RESULTS FOR WASTE FEED SAMPLES

The results of the analyses of waste feed samples for carbon tetrachloride and trichloroethylene by GC/ECD in the field and in the lab, and also by GC/MS in the lab, are shown in Table J-42 along with the expected concentrations based on the spiking rates and the waste feed flow rates.

The lab GC/MS results agreed most closely with the calculated spike concentrations. In most cases the laboratory GC/ECD results were considerably higher than the GC/MS results or calculated values. But, the field GC/ECD results were lower than the GC/MS results, except for CCl_4 in Run 4.

Some possible, but not confirmed, explanations for the observed results are the following:

- The high values for the samples analyzed by GC/ECD in the laboratory were probably due to interference by coeluting compounds. However, it is not possible to state which compounds may have coeluted because a different GC column with different retention order of the compounds was used for the GC/ECD analyses than for the GC/MS analysis.
- The low results by GC/ECD in the field may have been due to the fact that the sample concentrations were not within the linear range of the ECD detector. Thus, even though there were interfering compounds, the detector response of the compounds was too low.

ANALYSIS RESULTS FOR GAS BULB SAMPLES

A series of gas bulb samples of the stack effluent were taken during the eight test runs. These samples were analyzed by field GC/ECD, and six were returned to the analytical laboratory for analysis by GC/MS. The results of the analysis for CCl_4 and TCE are presented in Table J-43. The concentrations as determined by the VOST sampling method are also presented for comparison.

The data presented in Table J-43 show that the gas bulb samples generally gave much higher results than VOST, both for field GC/ECD and laboratory GC/MS. It must be noted that gas bulb samples are one-second grab samples whereas VOST samples are 20-min integrated samples, but it is not known why the gas bulb samples are always higher than VOST.

In Run 1 the syringe blank associated with field GC/ECD analysis of the gas bulb samples was relatively high and the same is true for TCE in Runs 2 and 3. The contamination may have been due to the presence of the liquid waste samples stored in the analysis area. The waste samples were removed in later runs.

TABLE J-42. COMPARISON OF RESULTS FOR DETERMINING LIQUID ORGANIC WASTE FEED ANALYSIS METHODS ($\mu\text{g/g}$)

Method ^a	Run 1		Run 2		Run 3		Run 4		Run 8A		Run 8B	
	CCl ₄	TCE	CCl ₄	TCE	CCl ₄	TCE	CCl ₄	TCE	CCl ₄	TCE	CCl ₄	TCE
Field GC/ECD	4,300	5,200	6,500	3,200	7,000	5,400	59,000	20,000	b	b	b	b
Lab GC/ECD												
1st Sample	24,000	16,000	24,000	16,000	24,000	16,000	50,000	41,000	30,000	23,000	21,000	15,000
2nd Sample	29,000	15,000	27,000	17,000	34,000	17,000	110,000	110,000	-	-	-	-
Lab GC/HS	10,000	8,400	10,000	6,800	9,900	10,000	33,000	35,000	12,000	15,000	11,000	14,000
Calculated ^c	9,400	8,500	9,100	8,300	11,300	10,300	28,000	25,400	13,000	12,000	11,000	10,000

GC/ECD = Gas chromatograph/electron capture device.

GC/HS = Gas chromatograph/mass spectrometer.

^a Each method used a different sample of the same waste feed.

^b This sample was not analyzed by field GC/ECD.

^c Calculated concentration, based on spiking rate and measured waste feed rate.

TABLE J-43. COMPARISON OF EFFLUENT CONCENTRATIONS DETERMINED BY DIFFERENT METHODS (ng/L)

		Carbon tetrachloride				Trichloroethylene			
		Gas bulb sample		Syringe blank		Gas bulb sample		Syringe blank	
		Lab GC/MS	Field GC/ECD	Field GC/ECD	VOST ^a	Lab GC/MS	Field GC/ECD	Field GC/ECD	VOST ^a
Run 1	A	b	5	4	1.0	b	63	48	3.0
	B	b	6	4	0.8	b	120	48	3.7
	C	b	3	4	d	b	47	48	d
Run 2	A	b	15	6	d	b	78	96	d
	B	b	340	6	d	b	310	96	d
	C	b	42	6	0.7	b	113	96	6.8
Run 3	A	1,100	1,400	13	d	110	67	23	d
	B	37	23	4	d	52	52	8	d
	C	10	9	1	1.3	36	43	18	4.2
Run 4	A	b	15	1 ^c	0.4	b	31	10 ^c	2.0
	B	4.8	30	1	4.0	25	63	10	1.8
	C	b	7	1	0.4	b	33	10	1.3
Run 8A		< 8	55	1	13	23	230	10	3.6
Run 8B		< 8	890	1	14	18	470	10	2.6

GC/ECD = Gas chromatograph/electron capture device.

GC/MS = Gas chromatograph/mass spectrometer.

^a This value is for the VOST trap which was in the stack during the time the gas bulb sample was taken. It is not blank corrected. For runs 1, 2, 3, and 4 this was a fast VOST sample, for runs 8A and 8B it was a slow VOST sample.

^b This sample was not analyzed by Lab GC/MS.

^c The same syringe blank was used for runs 4, 8A, and 8B.

^d This pair of traps was not analyzed.

There was relatively good agreement between the lab GC/MS and field GC/ECD analysis of the gas bulbs in Run 3, but in subsequent runs the field GC/ECD analysis gave significantly higher results than the lab GC/MS. The reason for this may be coelution of interfering compounds during the GC/ECD analysis, or due to quantification relative to an inappropriate liquid calibration standard.

It is concluded from this data that further work is needed before the GC/ECD can be utilized to advantage in the field. However, some changes that may improve future field analysis with the GC/ECD include the following:

1. The same GC column packing material should be used for the GC/ECD analysis as for GC/MS. (However, the same temperature program conditions need not be used.) In this way, the mass spec data will indicate whether there were any interferences during the GC/ECD analyses.

2. Since contamination of gaseous samples can occur so readily, it is important to keep the waste feed samples away from the GC/ECD analysis area. A separate room for analysis is a minimum requirement.

3. Methanol is probably a better solvent than hexane for diluting hazardous waste samples for analysis. Methanol will dissolve the relatively large amounts of water that are often present in hazardous waste as well as dissolve any of the volatile POHCs.

Methanol yields only a small sharp peak at the beginning of the GC/ECD chromatogram using the Carbowax column. Hexane does not yield a GC/ECD response at all, but does not appear to be as good a solvent to dissolve a total waste sample. Hexane was used as the solvent for the waste samples at TWI although methanol was used to dissolve the wastes during the preliminary GC/ECD lab analyses when some of the earlier waste samples were analyzed. The earlier analyses yielded results very similar to those from the GC/MS analysis of the same waste. Also, during the lab GC/ECD analysis, one of the waste samples was dissolved in triplicate in methanol rather than hexane. Analysis of the methanolic samples gave better precision than analysis of triplicate hexane extracts of the same sample (2.5% vs. 25% RSD for carbon tetrachloride and 6.1% vs. 30% RSD for trichloroethylene).

4. It is important that gaseous calibration standards be used to quantify gaseous components and liquid calibration standards be used to quantify liquid samples. Also, a wide range of calibration standard concentrations need to be prepared ahead of time so that each sample can be quantified relative to calibration standards that are analyzed at the same attenuation setting as the sample, and which bracket the sample concentration. One cannot assume that attenuation changes are linear. At TWI only one liquid calibration standard was used to quantify liquid and gaseous samples. The samples had to be analyzed at several different attenuation settings and it is doubtful that the linearity was adequate to accurately quantify the samples.

5. Gaseous samples are more troublesome to analyze than liquid samples. The glass bulbs for preparing calibration standards are bulky and it is necessary to dilute the gas from the initially prepared standard into another glass bulb to yield a gaseous standard dilute enough to avoid exceeding the detector's linear range. The gas-tight syringe used at TWI gave high blank values which complicated the analyses. The syringe contamination problem may be relieved by employing a relatively inexpensive commercial syringe cleaning device which uses a combination of heat and vacuum to remove organic contaminants. The sample should also be withdrawn from a separate septum port rather than through the stopcock.

Also, more work is needed to determine why laboratory GC/MS results for gas bulb samples are always significantly higher than VOST results.

DESCRIPTION OF SAMPLING AND ANALYSIS PROCEDURES

Gas Bulb Sampling Procedure

Gas bulbs were oven heated for 30 min at 150°C before use. After cooling, the bulbs were connected to the Teflon stack sampling line with a condensate preseparator. Stack gas was pulled through the 250 ml glass gas bulb by a pump, at a rate of about 15 L/min for 3 to 4 min. Stopcocks on each end of the gas bulb were then closed. The gas bulb was disconnected from the sampling line for analysis. This procedure yielded a nearly instantaneous (one-second) sample of stack gas.

Field GC/ECD Analysis Procedures for Liquid Waste Feed Samples and Gas Bulb Samples

The GC used was a Varian 3700 with a Ni-63 electron capture detector. The separation column was stainless steel, 3 m x 3.2 mm, containing 20% SP-2100/0.1% Carbowax 1500 on 100/120 mesh Supelcoport (EPA Method 23, halogenated organics). The column was operated isothermally at 55°C. When higher molecular weight compounds were expected in the samples, the temperature was programmed up to 250°C at 20°C/min after completion of the isothermal run. Manual injection was used with a 10 µl syringe for liquids and a 5 cc gas-tight syringe for gas samples.

Each sample was injected at least twice. Due to unexpected sample concentrations attenuation changes were sometimes made during the analysis. However, not all peaks were measurable. All peaks were compared against a single mixed standard of 8 pg/µL carbon tetrachloride and 7.3 pg/µL TCE (prepared by dilution from the 50/50 injector pump feed) in Burdick and Jackson n-hexane. All liquid injections were 1 µL and all gaseous injections were 1 cc. Peak areas were used for quantitation. It was not practical to prepare a gaseous standard in the field because of the difficulty in cleaning the gas tight syringe. No correction for temperature or pressure was applied to the data.

During Runs 1 and 2, the gaseous samples were found to have a high blank value associated with the syringe or the special long needle required to extend past the bulb stopcock. The blank values decreased with repeated use and depended on how the syringe was filled with cylinder nitrogen. The syringes were baked out and flushed with hexane with some success but a blank improvement was noted after the raw liquid waste feed samples were removed from the room at the start of Run 3. With only diluted liquid samples present in the area, the blank values were reduced. Blank values with the liquid syringe were always undetectable.

The ability to obtain a reproducible samples from the gas bulbs was found to depend upon equilibration time in the "warmer" laboratory. Once the bulbs had come to equilibrium, the long needled gas syringe was inserted into the bulb through the septum, the stopcock was opened and the syringe needle was further inserted into the bulb to its limit (3 to 4 in.), the plunger was pulled out to the 1 cc mark, and the syringe was allowed to set undisturbed for 5 min. The syringe valve was then closed and the syringe withdrawn from the bulb. The needle was replaced with a smaller gauge needle, and the sample was injected onto the GC column. Any variations from this routine resulted in imprecise peak areas. Short equilibration times and possible recontamination of ambient air caused problems during Runs 4 and 8.

Laboratory GC/MS Analysis Procedure for Gas Bulb Samples

Gas bulb samples returned to the analytical laboratory were connected with glass and teflon tubing, to a Tenax/charcoal VOST trap which had previously been analyzed and determined to be free of any contamination. Prepare N_2 was then pressured through the gas bulb to purge the contents of the bulb onto the trap. A N_2 flow of 100 ml/min was used to purge the bulb for 10 minutes. Thus, the total quantity of N_2 used was 1,000 ml, or 4 times the volume of the 250 ml gas bulb.

After purging the bulb with N_2 , onto the Tenax/charcoal trap, the trap was removed and analyzed by thermal desorption with GC/MS analysis using the same procedures as for all VOST traps.

Laboratory GC/ECD and GC/MS Analysis Procedures for Liquid Waste Feed Samples

Organic waste feed samples were returned to MRI and analyzed by GC/ECD in the laboratory. All wastes were diluted by a factor of 1:100,000 using hexane (B&J distilled in glass). Each 40-ml sample vial was shaken vigorously for 1 min and stirred with a glass pipette prior to aliquoting. Immediately, 0.5 g of the mixed waste was placed into a 100-ml volumetric flask and hexane was used to bring the volume to the mark. The flask was shaken vigorously for 1 min and then placed into a sonic bath for 1 min to ensure sample homogenization. One hundred microliters of the sample were drawn out and placed into a hexane-filled 50-ml volumetric flask. This final dilution was mixed for 1 min. Of each of the sample dilutions made, 1.0 μ l was analyzed by GC/ECD using a Supelco 20% SP-2100/0.1% Carbowax 1500 on 100/120 Supelcoport, 10 ft x 1.8 in. s.s. column. The GC/ECD analysis conditions were as follows:

Oven temperature: 55°C
Flow rate: 40 ml/min N₂
Det.: ECD No. 7
Injector temperature: 200°C

The GC analysis was conducted over 2.5 days. Calibration standard solutions at 0.1, 0.2, 0.5, and 1.0 ng/μL were analyzed before and after the samples. Calibration standards at the 5.0 and 10.0 ng/μL level were also analyzed; however, linearity was limited to the 0.1 to 1.0 ng/μL range. All sample concentrations were bracketed by this linear range of standards.

For most samples, triplicate injections were required to obtain accurate measurements. All concentrations were determined using linear regression of the calibration standards. In addition to triplicate injections, quality assurance measurements were also obtained by analyzing a re-extracted sample.

GC/MS analysis of waste feed samples was done according to the usual procedures as described in the body of this appendix for Runs 1-4.

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